CHEM 4B LAB: GENERAL CHEMISTRY FOR MAJORS II

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Chem 2BH: Laboratory Manual

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Licensing

A detailed breakdown of this resource's licensing can be found in ***Back Matter/Detailed Licensing***.

SECTION OVERVIEW

Preface and Lab Details

This manual is the culmination of the efforts of many individuals. While many of the experiments are "classics", and appear in various forms in many Quantitative Analysis textbooks and laboratory manuals, much effort was expended to ensure that the experiments work well here at UC Davis and thus each experiment has been extensively tailored for our laboratory program and equipment. Many faculty, teaching assistants, staff members, and students have been involved in this developmental process. While they are all too numerous to mention here, special acknowledgment must be given Ling Ng, for her efforts to continually improve these experiments for the benefit of the student. Also, for his revision of the library project, special thanks go out to Cory Craig of the Physical Science Engineering Library.

We view this manual as one of continual modification and over the past years many of these improvements have come from student comments and criticisms. We thus encourage you to discuss ideas for improvements with your TA and instructor. Special acknowledgements go to Dr. John Berg, Dr. Dara Gilbert, Kymton B.J. deCesare, Pam Lord, Heather Kalish, Debra Odink, John Sherlock, and Travis Ruthenburg.

A: Safety Policy

It is critical that you prepare for each experiment by reading it carefully before entering the laboratory. Not only will this ensure that you get the maximum benefit of the experience, but it also makes for a safer environment in the laboratory. This is important not only for your own safety but also for those around you. Several policies have been developed in order to make sure that the laboratory is safe and that it runs smoothly.

In each experiment, specific hazards are indicated by bold type and procedures are described that must be adhered to. In addition, accidents commonly occur when the following rules, as approved by the Chemistry Department Safety Committee, are not followed.

Student Name

Locker Number

Laboratory Information

Teaching Assistant's Name

Laboratory Room Number

Dispensary Room Number Chem Annex 3415

Location of Safety Equipment Nearest to Your Laboratory

Safety Shower

Eye Wash Fountain

Fire Extinguisher

Fire Alarm

Safety Chemicals

B: Laboratory Schedule

Assignments are in the Chemistry 4B Laboratory Manual. Students *must* read the assignment before coming to class. As a pre-laboratory preparation, the student must also write a title, purpose, brief outline of procedure, and outline of the data section in their bound notebook before beginning the experiment. **A student must complete all the assigned laboratory work in order to pass the course.**

Week	Starting Date	Exp #	Experiment Name	# Lab Period	Points
1	1/7	*Check-In & Safety*			
2	1/14	Exp 1	Thermochemistry (TC)	1	70
3	1/21	Exp 2	Standardization of Acids and Bases (SAB)	2	175
4	1/28				
5	2/4	Exp 3	Analysis of a Commercial Antacid (ACA)	1	50
6	2/11	Exp 4	Determination of the Molar Mass by Freezing Point Depression	1	100
7	2/18	Exp 5	Le Chatelier's Principle	1	100
8	2/25	Exp 6	Determination of K_c for a Complex Ion Formation II /Checkout	2	150
9	3/4				

C: Laboratory Policies

Goggles & Lab Coats

Students must wear OSHA approved safety goggles and white 100% cotton laboratory coat in the laboratory at all times. Failure to follow this rule will cause the student to be expelled from the laboratory. It is required that you wear clothing that completely covers your legs and feet while working in the laboratory. Inadequate protection often leads to injury. In addition, gloves will be provided for all experiments.

Pre-Laboratory Preparation

Many of the Chemistry 2BH laboratory experiments are quite long and many use chemicals that could present a hazard if used improperly. Thus, students are required to judiciously prepare for each experiment by carefully reading the experiment and writing a Title, Purpose, Procedure (brief outline), and Data (tables) section before arriving at the laboratory. Any student without these sections completed at the beginning of the period will have to leave the laboratory room until they are completed. In this situation, the student will still be required to complete the experiment in the allocated time and no extra time will be granted.

Writing a Laboratory Report

You will report your experimental findings in the form of a laboratory report. Below is the suggested format that your report should follow. The report should be written in your bound laboratory notebook. You will turn in an accurate high quality and easily readable photocopy of the report as it appears in the laboratory notebook at the beginning of the period following the one which was allocated for the completion of the experiment.

Below is a general outline of a common format that is often used in science laboratory courses. **Discuss this format with your T.A. during the first laboratory period so that you clearly understand what will be expected.**

- **Title**: The report should have a unique title which concisely describes the experiment. It should not be the title of the experiment.
- **Purpose**: This is a brief and concise statement which describes the goals of the experiment, the methods which are employed, and the major results of the experiment. Any pertinent chemical reactions are generally indicated.
- **Procedure**: A brief and concise outline (paragraph format) in your own words of each step of the experiment should be included. It should not be a copy of the lab manual If you are using a published procedure, you should also cite the literature or laboratory manual. A drawing of an unusual or hard to describe apparatus can also be included.
- **Data and Observations**: Report all measurements and observations which are pertinent to the experiment. Be sure to note any problems or unexpected occurrences. It is important that this section be as neat and as organized as possible. The use of tables will often help in this regard. **All data must be recorded directly into the notebook at the time it is collected.** Severe penalty will be imposed for pencil or transcribed data entries. Mistakes are not deleted. Simply draw a line through the error and record the correction. Your notebook is subject to examination at any time.
- **Calculations**: This section generally includes any complicated calculations which are involved in the experiment. Again it is important to use foresight when organizing this section. Equations are recommended to be left in terms of variables and plugging in the variable for each trial is not wanted.
- **Results**: Report the outcome of the experiment in table form and include the relevant statistics here. Brief interpretations of observations and results may also be discussed in this section.
- **Conclusions**: Discuss the outcome of the experiment and any consequences the results might have. Discuss any sources of error in the experiment and your confidence in the results. Describe any practical methods that could be used to improve the experiment.
- **Questions**: All assigned questions are answered in this section.

All reports must be typed. A date should be indicated on each report, especially in the Data section. You must prepare for each experiment by writing the Title, Purpose, Procedure, and blank data tables before coming to the laboratory. It is also important to organize and prepare the format of the Data section before coming to the laboratory so that you will only need to neatly record your data and observations during the experiment. Each section should be clearly marked with a proper heading. Your notebook should be organized and written in such a manner that another chemist could read it and repeat the experiment in precisely the way in which you did it.

Laboratory Make-Ups

Because many of the experiments require standard solutions and/or solutions of a limited shelf life, it is absolutely essential that students make every effort to come to the proper laboratory period to complete the experiment in the allocated time. In cases where the student cannot attend their registered laboratory period (emergencies and sickness only) **the student must supply verification of the emergency to their TA. The TA will then write a letter of exception that the student can use to gain admittance to another upcoming laboratory period.** Students will not be allowed to work in a non-registered laboratory period without such a letter. **Only in verified cases will the TA write such a letter and the student must make-up the experiment as soon as possible.** It is the student's responsibility to notify the stockroom of their intention to make-up an experiment so that all the needed chemicals are prepared.

Late Reports

Students must turn in their reports at the beginning of the laboratory period which follows the one allocated for the completion of the experiment. Students will turn in a typed laboratory report with copies of their original data attached. The reports will be promptly graded and returned to the students by the teaching assistant.

Late reports will incur a 5-point deduction for every day the report is late.

Unknowns

Students will obtain all unknowns from the stockroom (Room 3415). You must be explicit in your request for an unknown; that is, be sure you know the name of the experiment and unknown. You are required to show your laboratory notebook to the stockroom assistant who will mark in your book indicating that an unknown was obtained. A student may obtain extra unknown only once during the quarter without deduction. Any later requests for extra unknown will be met with a 50% reduction in points for that experiment. Extra unknown will only be provided to allow a student to complete the experiment and will not be provided to allow the student to refine result.

1: Thermochemistry (Experiment)

Introduction

This experiment is an introduction to the basic principles of thermochemistry and involves the exchange of energy as heat. The ideas and concepts involved in thermodynamics are in your everyday experiences. For example, on a hot summer day, the hood of a car can get hotter than the sidewalk cement, and when cooking, you have probably noticed that a wooden spoon does not heat as fast as a metal one. After completing this lab, you will better understand the reasons behind these and other thermal phenomena.

Safety Precautions

WEAR SAFETY GOGGLES throughout the entire experiment. To avoid burns, use crucible tongs to pick up hot metal. Never pick up a heated metal with your bare hands. All waste from this experiment can be poured down the drain.

Part I: Determining the Heat Capacity of the Calorimeter

All parts of this experiment require the use of a calorimeter. In this part of the experiment, you will construct an inexpensive but effective coffee-cup calorimeter. For this experiment, we will consider the calorimeter to be a perfect insulator and for it to have no heat capacity.

The heat capacity of a substance is the heat required to raise the temperature of one gram of the substance one degree Celsius or Kelvin. All substances have a characteristic heat capacity. When two substances having different temperatures come into contact, energy in the form of heat is exchanged between them until they reach a common temperature. If they are insulated from their surroundings, the amount of heat lost from the hotter substance equals that gained by the colder one. The heat lost or gained is related to the mass, the heat capacity of the substance, and the temperature change. This relationship is shown below:

$$Q = m \times C_p \times \Delta T \tag{1.1}$$

where Q is the heat, m is the mass, C_p is the heat capacity of that substance, and ΔT is the change in temperature.

Procedure

1. Set up the hot plate and heat 500 mL of deionized water to boiling in a 600 mL beaker.
2. Put two Styrofoam coffee cups in a 250 mL beaker.
3. Place a 4"x 4" piece of cardboard, with the hole in the center, on top of the coffee cups. If the hole is too big, cover it with two pieces of lab tape and punch a hole with your pen or pencil big enough so the temperature probe can fit. Insert a temperature probe through the hole.
4. Take the top Styrofoam cup from the calorimeter and tare it. Weigh out about 100 grams of deionized water into it and record the mass.
5. Place the cup back into the calorimeter set-up and cover with the cardboard top. Using a clamp, gently secure the temperature probe and lower it into the cup so that the tip of the probe is covered with water but is not touching the bottom of the cup. Allow the temperature probe to sit in the water for a few minutes before recording the temperature.
6. Obtain a sample of lead from your TA. The sample should be strung with nylon string. Do not remove the string. Weigh the lead sample on a balance, using weighing paper to protect it from contamination. Record the mass.
7. Suspend the lead metal into the beaker of boiling water by putting a glass stirring rod through the loop of the nylon string and placing the stirring rod across the top of the beaker. Make sure the metal is completely submerged in the boiling water and is not in contact with the sides of the beaker. Suspension of the sample ensures that the metal will have the same equilibrium temperature as the water by preventing direct heating from the hot plate. If the metal is in direct contact with the glass, the glass may shatter.
8. When the water begins to boil, allow the metal to remain in the boiling water for 2-3 minutes. This will ensure the temperature of the metal equilibrates with the temperature of the boiling water.

Question 1.1

Why is the temperature not expected to be exactly 100 °C? Explain.

9. Measure the temperature of the boiling water bath using the **temperature probe**. Start data collection on the LabQuest device. Record the temperature for 1 minute, then cool and dry the temperature probe.
10. When the metal is ready to be transferred, start data collection on the Logger Pro device and measure the temperature of the water in the calorimeter using a temperature probe. Measure the temperature for 1 minute.
11. Finally, begin data collection and then lift the metal from the hot water with the nylon string slightly slanted so water will drip off the metal. Remove the temperature probe and cardboard top from the calorimeter and then quickly drop the metal into the calorimeter and cover with the cardboard with the temperature probe. Make sure the metal is completely covered with water.

Question 1.2

Why do we want the water to drip off the metal before it is placed in the calorimeter?

12. Adjust the temperature probe's height so that it is not touching the lead or the Styrofoam cup, yet the water is covering the entire temperature probe bulb.
13. Gently swirl the calorimeter to distribute the heat throughout the calorimeter. Watch the Logger Pro display closely as the temperature rises. Sometimes it will rise, then fall, and rise again due to the uneven distribution of heat within the calorimeter. Record the temperature for a two-minute time period.
14. Repeat this procedure two more times. Dry the calorimeter and temperature probe between trials.
15. Calculate the heat capacity of your calorimeter. Report the average heat capacity for the calorimeter. Also, calculate the standard deviation, 95% confidence limit, and relative deviation of your data.

Part II: Determining the Heat Capacity of a Metal

In this experiment, you will determine the heat capacity of a metal using the coffee cup calorimeter you constructed in Part I of the experiment.

Procedure

1. Using your calorimeter for which the heat capacity has now been determined, you will analyze an unknown metal sample by essentially repeating the procedure in Part I. The sample will be either **aluminum** or **copper**. (Remember to record the color of your unknown sample.)
2. Calculate the specific heat for your metal for each run. (Remember that the heat lost by the metal is equal to the heat gained by the water in the calorimeter and by the calorimeter itself.) Calculate the average specific heat for your metal as well as the standard derivation, rel. deviation, the 95% confidence limit of your data. Then, using the values given, determine the identity of the metal and then calculate the percent deviation of your calculated specific heat value from the accepted value. (Al = 0.900 J/g·C; Cu = 0.389 J/g·C)

Question 1.3

Do you expect your value for the specific heat to be too high or too low? Explain.

Question 1.4

Although the coffee-cup calorimeter is efficient, it is not perfect. It does absorb some heat. How would this affect your results?

Part III: Calculating the Enthalpy of an Endothermic Reaction

The cold packs in some first-aid kits are made of ammonium nitrate pellets encased in a plastic bag surrounded by water. When the cold-pack is bent, the inner bag is broken, and an endothermic reaction occurs as the ammonium nitrate dissolves in the water. You will be simulating this reaction in this part of the experiment.

Procedure

1. Remove the cardboard top of your calorimeter. Accurately measure about 100 mL of water and place it into the calorimeter. Record the temperature of the water for one minute.
2. Accurately weigh out approximately 20 g of ammonium nitrate and record this mass in your notebook.

3. Add the ammonium nitrate to the water in your calorimeter. Stir the reaction mixture with a stirring rod. Begin data collection and record the temperature for at least two minutes.
4. Determine the accurate temperature change based on your measurements.
5. Using the molecular mass of ammonium nitrate and assuming all the heat is absorbed by the water in the calorimeter (4.184 J/g·C), calculate the enthalpy of the reaction per mole of ammonium nitrate.

Question 1.5

Why does a cold pack "feel" cold?

Part IV: Calculating the Heat of Fusion of Water

Now is your chance to design an experiment. This is an especially important experience since many of you will be pursuing careers in fields that require you to do research. In this part of the experiment, you will design an experiment to determine the heat of fusion of ice. You will probably need your calorimeter, ice, water, and a balance. You may use any of the equipment in your locker. Be sure your method is repeatable. Have fun!

Procedure

1. Design an experiment to determine the heat of fusion of ice.
2. Do the experiment. Write up the procedure.
3. Calculate the heat of fusion from your data, and then calculate the relative error of your value for the heat of fusion relative to the accepted value in your text.

Clean Up

All ammonium nitrate solutions must be disposed of in the CHEM 4B Exp. 1 – Thermochemistry waste container. Be sure to rinse out the calorimeter before returning it.

2: Standardization of Acids and Bases (Experiment)

Introduction

This experiment affects several experiments that follow. You should strive for maximum precision in this experiment since inaccuracies here will result in inaccuracies later.

Safety Precautions

Be especially careful when using the strong acid or strong base solutions as they can cause severe burns. Since concentrated HCl has a pungent odor it would be wise to dispense this solution in the hood. All waste solutions may be disposed of by rinsing them down the drain.

Part I. **Preparation of Solutions**

1. An approximately 0.1 M NaOH solution will be prepared by dilution of a 50 weight-percent NaOH solution. This solution has a density of 1.53 g/mL. Compute the volume of this solution that is required to prepare 1000 mL of 0.1 M NaOH. If you are unsure of your answer, confirm it with your TA.

2. Obtain 1000 mL volumetric flask, add around 400-500 ml (halfway) of deionized water from the proper carboy in the laboratory. Using the 50% by mass stock solution, measure the calculated volume of NaOH solution into a clean, dry 10 mL graduated cylinder.

3. Pour the contents of the graduated cylinder into your flask. Fill the graduated cylinder with five successive 10 mL portions of deionized water, each time emptying it into the flask.

4. Swirl then add more deionized water until the final volume of the system is 1000 mL. Pour the contents into the 1 L storage bottle and cap the bottle.

5. Label the bottle with chemical name and concentration. Allow the solution to return to room temperature. Sodium hydroxide solutions should never be left uncapped. Base solutions will absorb carbon dioxide from the atmosphere, thereby changing the titer of the solution.

Question 2.1

Give the balanced chemical equation for the formation of carbonic acid and for the reaction of carbonic acid with sodium hydroxide.

6. An approximately 0.1 M HCl solution will be prepared by dilution of concentrated HCl, which is about 12 M. Compute the volume of this solution that is required to prepare 1000 mL of 0.1 M HCl. If you are unsure of your answer, confirm it with your TA. **DO NOT REMOVE THE CONCENTRATED HCl BOTTLE FROM THE FUME HOOD!**

7. Obtain 1000 mL volumetric flask, add around 400-500ml (halfway) of deionized water from the proper carboy in the laboratory. Measure the calculated volume of concentrated HCl solution into a clean, dry 10 mL graduated cylinder. Pour the contents of the graduated cylinder into your flask.

8. Always remember to add acid to water and never the reverse. Swirl then add more deionized water until the final volume of the system is 1000 mL. Pour the all the contents into the 1L storage bottle and cap the bottle. Label the bottle with chemical name and concentration. Allow the solution to return to room temperature.

Question 2.2

Why do we always add acid to water and never the reverse?

Part II. Standardization of the NaOH Solution

1. (Please note: KHP has already been dried in the oven for your convenience). Label three 125 mL Erlenmeyers Flasks (ensure that they are cleaned and dried.)

2. On a weigh boat, weigh out approximately 3 grams of KHP, and record the value. Keep the KHP and weigh boat on the balance.
3. From this mass of KHP, take about 0.7-0.9 gram of material and transfer it to one of the Erlenmeyer Flasks. Record the new mass of KHP on the weigh boat. The difference in masses is the mass of KHP in your Erlenmeyer Flask.
4. Repeat step 3 for the remaining Erlenmeyer Flasks. This process of weighing starting material is weight by difference.
5. To each of the Erlenmeyer flasks, add 50-75 mL of deionized water and 2 drops of phenolphthalein indicator solution. Against a white background, titrate each flask with your NaOH solution to the first appearance of a faint pink color that persists for 30 seconds. You will have to learn to "split" drops to get a very faint pink endpoint. Tip: you should use a black-lined background card to accurately read the meniscus.
6. Wash down the sides of the flask with deionized water using your water bottle to be sure all the reactants are in the solution.
7. Given that the molecular weight of KHP is 204.23 and that it is a monoprotic acid, calculate the molarity of NaOH for each of your samples. Your triplicate molarities should agree to within three parts per thousand. Calculate the average, standard deviation, 95% confidence limit, and relative deviation. This is easily done on a computer spreadsheet.
8. **Carefully cap and store the solution for use in later experiments.**

Question 2.3

Why does it not matter how much water you add when dissolving the acid or when carrying out the titration?

Part III. Unknown KHP Analysis

You will be given an unknown sample of KHP (KHC8H4O4). Analyze the sample in the same way that you standardized your NaOH solution. A brief outline of the procedure is described below.

1. Label three 125 mL Erlenmeyers Flasks (ensure that they are cleaned and dried.)
2. (An assigned unknown will be provided by your TA). Weigh by difference three samples of your dry, cooled unknown KHP into three separate 125 mL Erlenmeyer Flasks.
3. On a weigh boat, weigh out approximately 3.3 grams of unknown KHP, and record the value. Keep the KHP and weigh boat on the balance.
4. From this mass of KHP, take about 0.9-1.1 grams of material and transfer it to one of the Erlenmeyer Flasks. Record the new mass of KHP on the weigh boat. The difference in masses is the mass of KHP in your Erlenmeyer Flask.
5. Repeat step 4 for the remaining Erlenmeyer Flasks.
6. Dissolve in deionized water and titrate with standard NaOH.
7. Calculate the mass percent of KHP in each sample. Report an average, standard deviation, 95% confidence limit, and relative deviation.
8. **This is a good stopping point for day 1 of the lab. Please clean all glassware and report to TA before leaving.**

Question 2.4

Write a balanced chemical equation for this reaction.

Part IV. Standardization of HCl Solution

This is a more complicated procedure than the one you have just accomplished. The reason for this is that there are very few primary base standards. One of the best is sodium carbonate. However, the use of this standard is complicated by the fact that carbonate is a weak acid and that a final product of the titration is a gas. The two acid/base reactions are:

$$\mathrm{H^+ + CO_3^{2-} \rightleftharpoons HCO_3^-} \tag{2.1}$$

$$\mathrm{H^+ + HCO_3^- \rightleftharpoons H_2CO_3 \longrightarrow H_2O + CO_2(g)} \tag{2.2}$$

These two reactions combine to from a solution which is called a buffer and which is described in Chapter 10. We will boil the solution near the end of the titration to drive out the carbon dioxide and thus destroy this buffer and get a much sharper endpoint.

Procedure

1. After the Na_2CO_3 has dried for at least 1 hour, remove the beaker from the oven using tongs, and set it on a pad of paper on the bench top.

2. Allow it to cool there until you can put the inside of your forearm against the beaker without discomfort. Then put the beaker into a desiccator and allow it to cool for at least another half-hour. The weighing bottle should remain uncapped throughout this operation.
3. Label three 125 mL Erlenmeyers Flasks (ensure that they are cleaned and dried.)
4. On a weigh boat, weigh out approximately 0.8 grams of $\mathrm{Na_2CO_3}$, and record the value. Keep the $\mathrm{Na_2CO_3}$ and weigh boat on the balance.
5. From this mass of $\mathrm{Na_2CO_3}$, take about 0.2 to 0.25 grams of material and transfer it to one of the Erlenmeyer Flasks. Record the new mass of $\mathrm{Na_2CO_3}$ on the weigh boat. The difference in masses is the mass of $\mathrm{Na_2CO_3}$ in your Erlenmeyer Flask.
6. Repeat step 5 for the remaining Erlenmeyer Flasks.
7. Dissolve the first sample of sodium carbonate in 50 mL of deionized water and add 20 drops of bromocresol green indicator. Titrate the solution with HCl until the solution *just* changes from blue to green.
8. Wash down the sides of the flask with deionized water using your water bottle to be sure all the reactants are in the solution. Boil the solution for 2 to 3 minutes, during this time the color should revert back to blue. If it does not, then a measured amount of base must be added to change the color to blue and this volume of added base must be included in the calculations. Cool to room temperature, and complete the titration to the final green endpoint.

Question 2.5

What is the reason for boiling the solution? Why is it important?

6. Compute the HCl molarity. Report an average, standard deviation, 95% confidence limit, and relative deviation.

Question 2.6

Write balanced equations for the reactions involved in this standardization.

7. **Carefully store and cap the solution for use in later experiments.**

Part V. Unknown Soda Ash Analysis

Obtain an unknown sample of soda ash from the stockroom. Soda ash is a mixture of sodium carbonate and an unreactive substance. Analyze the sample in the same way that you standardized your HCl solution. A brief outline of the procedure is described below.

1. Label three 125 mL Erlenmeyers Flasks (ensure that they are cleaned and dried.)
2. On a weigh boat, weigh out approximately 1.8 grams of unknown soda ash, and record the value. Keep the unknown soda ash and weigh boat on the balance.
3. From this mass of unknown soda ash, take about 0.56 to 0.60 grams of material and transfer it to one of the Erlenmeyer Flasks. Record the new mass of unknown soda ash on the weigh boat. The difference in masses is the mass of unknown soda ash in your Erlenmeyer Flask.
4. Repeat step 3 for the remaining Erlenmeyer Flasks.
5. Dissolve in deionized water and titrate with standard HCl.
6. Calculate the mass percent of sodium carbonate in each sample. Report an average, standard deviation, 95% confidence limit, and relative deviation.

Clean-up

After the experiment is completed, drain any remaining solution from the burette. Rinse each burette with deionized water. Then, fill the burette with deionized water and carefully place it in the burette rack.

Helpful hint

You will use your burette in many of the Experiments in Chemistry 4B. **It is important that you always clean it at the end of the day and fill it with deionized water before storage**. It is also important that when you use a burette that you follow the recommended procedure for changing a solution. First empty the burette out the top and half-fill it with deionized water. Open the stopcock and drain ~5 mL out the tip. Empty the burette out the top and repeat the water washing, this time also opening the stopcock when the burette is inverted to drain most of the water from the tip. Wait ~30 seconds for drainage and close the stopcock.

Add the new solution to about the 48 mL mark. Drain some of it out the tip and close the stopcock. At the sink, cradle the top of the burette between the thumb and index finger of one hand and turn the burette horizontal. Then twirl the burette and slowly empty it through the top, being careful to wet the entire interior wall with the new solution. Repeat this operation two more times. Finally, fill the burette above 0.00 mL mark and drain the excess out the tip until a reasonable starting mark is reached. Follow this procedure whenever you change the solution in a burette.

3: Analysis of a Commercial Antacid (Experiment)

Introduction

In this experiment, you will use the standard acid and standard base that you prepared earlier to analyze the neutralization capacity of a commercial antacid.

Safety Precautions:

Be especially careful when using acid or base solutions as they can cause severe burns. All waste solutions may be disposed of by rinsing them down the drain.

Procedure

1. Obtain an unknown antacid sample from the front counter. Using a mortar and pestle, grind the sample to a fine powder. To efficiently do this experiment you will first do a quick titration to get a rough idea of the neutralization capacity of your unknown. Your class will then be divided into three groups. You will be assigned to conduct three runs of accurate titrations on one of the three commercial antacid brands provided. You will share your data with the rest of the class after the experiment. Meanwhile, data on the other two antacids will be made available for you after everyone is done with their calculations.
2. Into a 300 mL beaker weigh, by difference, one sample of the unknown (weigh to ± 0.1 mg) within the mass range of 0.1600 to 0.2500 gram. Add 50 mL of your standard 0.1 M HCl solution using your 25 mL pipet. Swirl the beaker to wet the samples thoroughly with acid, and allow them to stand for about 15 minutes. Then set the hot plate to very low, around 20% heating intensity, with occasional swirling (with tongs!), for about 20 minutes. At the end of the heating period, add three drops of phenolphthalein indicator. If the contents turn pink, add another 25.00 mL aliquot of standard acid and continue heating for about 15 minutes. Antacid tablets often contain water-insoluble binders so your solution may look cloudy. This should not adversely affect your results.

Question 3.1

Write a balanced chemical equation for this reaction.

3. Remove the beaker from the heat source and cool it to room temperature using a dishpan of water, being careful not to contaminate the contents of the beaker. Just prior to titrating your sample, add about 5 drops of phenolphthalein indicator to the solution and titrate to the visual endpoint with your standard 0.1 M $NaOH$ solution. Wash down the sides of the beaker with deionized water using your water bottle to be sure all the reactants are in the solution. The indicator will change colors from colorless to pink. This is the same as the first endpoint in the carbonate titration. For reasons of accuracy, it is important that the volume of $NaOH$ required to be between 25 mL and 45 mL. If your volume is not within this range, you will have to adjust the mass of antacid sample for the next analyses. Using the information gained from this rough titration you are now ready to make an accurate determination.
4. Into three separate 300 mL Erlenmeyer flasks weigh, by difference, triplicate samples of the tablets of the unknown use the same weight as you used in the preliminary run (weigh to ± 0.1 mg). To each flask add 50.00 mL of your standard 0.1 M HCl solution using your calibrated 25 mL pipet. Swirl the flask to wet the samples thoroughly with acid, and allow them to stand for about 15 minutes. Then heat the flasks, with occasional swirling (tongs!), for about 20 minutes. At the end of the heating period, add three drops of phenolphthalein indicator to each flask.
5. Cool the flasks to room temperature under running tap water, being careful not to contaminate the contents. Titrate your sample, to the visual endpoint with your standard 0.1 M $NaOH$ solution. The repeat titrations will go much faster once the endpoint is found.

Calculations

1. Report the effective mass percent of OH^- or CO_3^{2-} for the antacid you measured. For remote students, just calculate it for CO_3^{2-}. The effective mass is the mass of OH^- or CO_3^{2-} that would be in the unknown if that were the only base present. The antacid samples actually contain a variety of bases.
2. Report the average percent by mass of OH^- or CO_3^{2-} in your unknown along with a standard deviation, a 95% confidence limit, and a relative deviation. This can easily be done on a computer. or remote students, just calculate it for CO_3^{2-}.

Question 3.2

Do you have more confidence in your analysis of the KHP or of the antacid? Why?

Question 3.3

Stomach acid is about 0.12 M HCl. Does your unknown consume 47 times its own weight of 0.12 M HCl solution? Show by calculation.

Clean-up. After the experiment is completed, drain any remaining solution from the burette. Rinse each burette with deionized water. Then, fill the burette with deionized water and carefully place it in the burette rack.

Post-lab exercise. The data from your laboratory section will be collected. From this information, determine the average effective mass percent $\mathrm{OH^-}$ or $\mathrm{CO_3^{2-}}$ for each type of tablet along with the standard deviation, 95% confidence limit, and a relative deviation. Based on this data, what product would you recommend to the consumer? Explain.

4: Determination of the Molar Mass by Freezing Point Depression (Experiment)

Objectives

- To determine the molar mass of an unknown solid using the colligative property of freezing point depression.

When a small amount of non-volatile solute is dissolved in a volatile solvent, the vapor pressure of the solvent over the solution will be less than the vapor pressure of the pure solvent at the same temperature. Therefore, the temperature at which the equilibrium vapor pressure reaches atmospheric pressure is higher for the solution than for the pure solvent. As a result, the boiling point of the solution, T_b, is higher than the boiling point of the pure solvent, T_b^o. The amount by which the boiling point of the solution exceeds the boiling point of the pure liquid, $\Delta T_b = T_b - T_b^o$, is called the **boiling point elevation**. Similarly, because of the reduction in vapor pressure over the solution, the freezing point of the solution, T_f, is lower than the freezing point of the pure solvent, T_f^o. The amount by which the freezing point of the solution is decreased from that of the pure liquid, $\Delta T_f = T_f^o - T_f$, is called the **freezing point depression**. Figure 4.1 below illustrates this behavior for water.

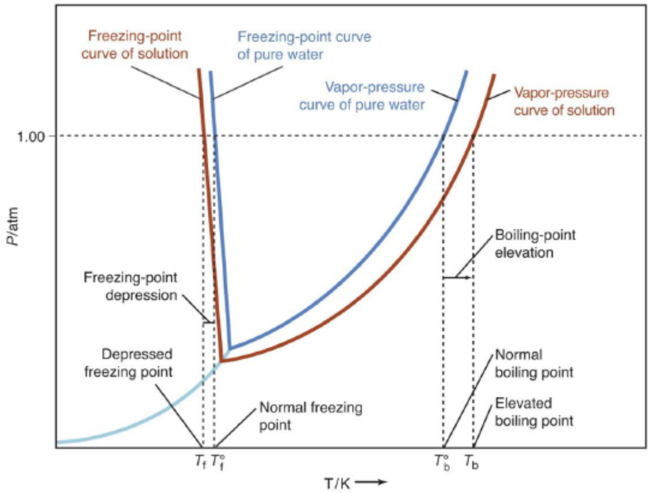

Figure 4.1

The magnitude of the freezing depression produced by a solute is proportional to its colligative molality, m_c:

$$\Delta T_f = T_f^o - T_f = K_f \times m_c \tag{4.1}$$

K_f is known as the **freezing point depression constant** and depends on the solvent used.

In this experiment, you will determine the molar mass of an unknown solid by dissolving a pre-weighed sample in a solvent and measuring the resulting freezing point depression of the solvent. From the measured ΔT_f and the known K_f value of the solvent, you can then determine the value of m_c using the above Equation 4.1. The colligative molality, m_c, is related to the molality of the solution, m, by the expression:

$$m_c = i \times m \tag{4.2}$$

where i is the number of solute particles produced per formula unit of dissolved solute, and m is the number of moles of solute per kilogram of solvent. Since only non-dissociating solutes will be used in this experiment, the value of i for your unknown solute can be considered to be 1. Thus, you may assume that $m_c = m$. From the experimentally determined value of m and the mass of solute added, you can determine the molar mass of the unknown solute.

The solvent that will be used in this experiment is para-dichlorobenzene, shown in Figure 4.2 below. Para-dichlorobenzene has a K_f value of 7.10 °C·kg·mol^{-1}. It also has a convenient freezing point that is near 60 °C.

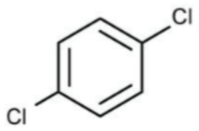

Figure 4.2

In order to determine the freezing point of this pure solvent, you must first heat it in a test tube to over 60 °C using a hot water bath, and then measure the temperature as a function of time as the liquid cools. At first, the temperature will fall quite rapidly. When the freezing point is reached, solid will begin to form, and the temperature will tend to hold steady until the sample is all solid. This behavior is shown in Figure 4.3 below. The freezing point of the pure liquid is the constant temperature observed while the liquid is freezing to a solid.

The cooling behavior of a solution is somewhat different from that of a pure liquid, also shown in Figure 4.3. As discussed earlier, the temperature at which a solution freezes is lower than that for the pure solvent. In addition, there is a slow, gradual fall in temperature as freezing proceeds. The best value for the freezing point of the solution is obtained by drawing two straight lines connecting the points on the temperature-time graph. The first line connects points where the solution is all liquid. The second line connects points where solid and liquid coexist. The point where the two lines intersect is the freezing point of the solution.

Note that when the solid first appears, the temperature may fall below the freezing point, but then it comes back up as more of the solid forms. This effect is called supercooling and is a phenomenon that may occur with both the pure liquid and the solution (Figure 4.3). When drawing the straight line in the solid-liquid region of the graph, ignore points where *supercooling* is observed. To establish the proper straight line in the solid-liquid region, it is necessary to record the temperature until the trend with time is smooth and clearly established.

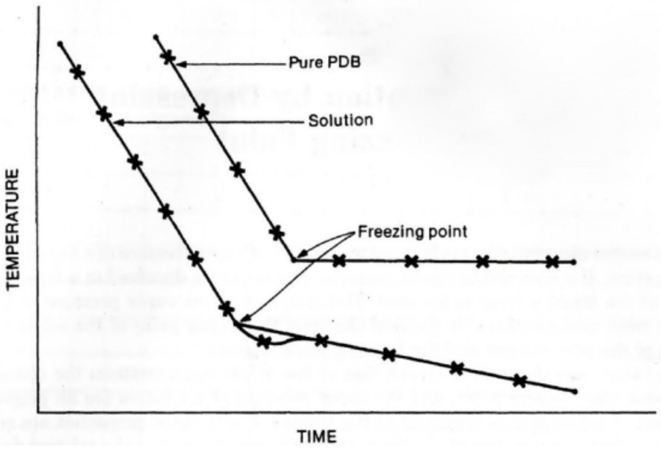

Figure 4.3

Procedure

Chemicals: Para-dichlorobenzene (PDB), unknown sample, acetone

Equipment: 600-mL beaker, 100-mL beaker, large test tube, utility clamps, hot plate, LabQuest with temperature sensor, thermometer, 1/2 inch magnetic stir bar

Safety

- The PPE for this lab includes safety goggles, lab coats, and nitrile gloves.
- Para-dichlorobenzene is used in mothballs and urinal cakes, and so it may have a familiar smell. However, direct inhalation of its vapors may be harmful or even toxic. Students should avoid skin contact with para-dichlorobenzene and direct inhalation of its vapors. All heating of para-dichlorobenzene must be done under a fume hood.

Waste Disposal

All chemicals used must be disposed of in the proper waste container. The acetone and para-dichlorobenzene must not go down the sink!

Part 1: Determining the Freezing Point of Pure PDB

1. Take note of the setup, which will be shown by the TA.

2. Fill a 600-mL beaker with tap water to the 500-mL line. Place the beaker of water onto the hot plate. This will serve as a hot water bath for the experiment. Use a thermometer to monitor the temperature of the water bath. Begin warming the water while setting up the rest of the experiment, but do not allow the temperature to rise too much above 65°C.
3. Weigh a clean, dry large test tube in a 100-mL beaker using an electronic balance and record its mass. Use a weighing boat and weigh out about 30 grams of para-dichlorobenzene (PDB). Slowly add it to the test tube (USE A FUNNEL TO AVOID SPILLS). Reweigh and record the mass of the test tube and the PDB. Calculate the mass of PDB in the test tube by difference. If there are spills of PDB around the balances, clean them up immediately! Use a brush and a weighing boat to collect the crystals, then dispose of them in the waste carboy.
4. Add a 1/2 inch magnetic stir bar, then use your utility clamp to attach the large test tube containing the PDB to the stand. Do not force the temperature sensor in the test tube. Ask the TA to double-check your lab setup.
5. **Setting up the LoggerPro:** Run Logger Pro from your desktop. Make sure the temperature sensor and LabQuest are both plugged in and working. Click on "experiment", then data collection setup. Make the following changes: Mode should be time-based; Rate 1 sample/s; Interval 1s/sample; Duration 420s. Click "done."
6. Heat the water bath gently, allowing the temperature to rise slowly to around 65°C. As the temperature of the water bath reaches the melting point of the PDB, it will begin to melt. After most of the solid PDB appears to have melted, insert the temperature sensor into the PDB and adjusting the top clamp. Adjust the bottom of the sensor so that it is about 1-2 cm above the bottom of the large test tube. Start the stirring on the hotplate; it should slowly melt any remaining solid PDB.
7. When the temperature of the PDB reaches about 65 °C, stop heating. Power off the hot plate and carefully lift the clamp with the test tube to remove the water bath. Slowly lower the clamp a few inches, so the stirring function will still work (at least 3-4 inches of space between the test tube and the hot plate.)
8. Monitor the temperature of the PDB as it cools. Stir the liquid slowly (using magnetic stirring on the hotplate) but continuously to help minimize supercooling. When the temperature of the PDB reaches 62 °C, begin recording its temperature by pressing on the **collect** button on the logger pro. Continue until the liquid has solidified to the point that you are no longer able to stir it. Stop the LabQuest by pushing the stop or collect button. Near the melting point, you will observe crystals of PDB in the liquid, and these will increase in amount as the cooling proceeds. Note the temperature at which these crystals first start to appear. Click on the "***Experiment***" tab in Logger Pro then click ***Store latest run*** to store your data and clear the graph.

Part 2: Determining the Freezing Point of PDB with about 2 g Unknown Solute

1. Your TA will give out instructions on computing for the weight of the unknown. Once you have made the calculations, carefully transfer about 2 grams of the unknown into the large test tube, taking great care that none of the unknown samples is spilled during this process. If you do spill some, you will need to start this step over with a fresh sample of weighed PDB.
2. Lower the large test tube to the hot water bath and heat the PDB-unknown mixture until it is completely melted. Stir well to mix the unknown with the PDB thoroughly. When the temperature of the mixture reaches 65°C, stop heating. Remove the hot water bath as before, and dry off the outside of the test tube with a paper towel.
3. Monitor the temperature of the PDB-unknown mixture as it cools. Stir the liquid slowly but continuously to help minimize supercooling. When the temperature of the mixture reaches 60°C, begin recording its temperature by pressing the collect button. Continue until the mixture has solidified to the point that you are no longer able to stir it and stop the Logger Pro by pushing the stop or collect button. Note the temperature at which crystals first start to appear. Click on the "***Experiment***" tab in Logger Pro then click ***Store latest run*** to store your data and clear the graph.
4. The dependence of temperature on time for the mixture should be similar to that observed for the pure PDB, except that the first crystals should appear at a lower temperature for the mixture. There may be some supercooling, as evidenced by a rise in temperature shortly after the first appearance of crystals.

Part 3: Determining the Freezing Point of PDB with about 4 g Unknown Solute

1. Prepare another 2 grams of the unknown sample and carefully transfer it into the large test tube, taking great care that none of the unknown samples is spilled during this process. If you do spill some, you will need to start this step over with a fresh sample of weighed PDB and about 4 grams of unknown.
2. Raise the hot water bath around the large test tube and heat the PDB-unknown mixture until both the PDB and unknown solid are completely melted. Stir well to mix the additional unknown with the PDB thoroughly. When the temperature of the mixture reaches 60°C, stop heating. Remove the hot water bath as before, and dry off the outside of the test tube using a paper towel.
3. Monitor the temperature of the PDB-unknown mixture as before. When the temperature of the mixture reaches about 57°C, begin recording its temperature by pressing the collect button. Continue until the mixture has solidified to the point that you are no longer able to stir it. Stop recording with Logger Pro and click on the "***Experiment***" tab in Logger Pro then click ***Store latest***

run to store your data. Note the temperature at which crystals first start to appear. Once again, some supercooling might occur. You can now export your data as CSV for your report.

Clean Up

1. When you have completed the experiment, melt the PDB-unknown mixture as before and then pour the warm liquid quickly into the waste container. Rinse any remaining residue from the side of your test tube, thermometer, and stirrer directly into the waste container using the small squirt bottle of acetone provided. Try to use as little acetone as possible, but be certain that all of the PDB has been removed before replacing the glassware in your locker. Do not allow any of the acetone or PDB-unknown mixture or rinse to go down the sink.
2. Wipe your area clean and return the glassware that you used to the appropriate bins.

4.P: Determination of the Molar Mass by Freezing Point Depression (Pre-Lab)

1. Camphor melts at 179.8^{o}C and has a freezing point depression constant, K_f = 40 ^{o}C/molal. When 0.186 g of an unknown organic solid is dissolved in 22.01 g of liquid camphor, the freezing point of the mixture is found to be 176.7^{o}C. What is the molar mass of the solute?
2. A particular unknown solid depresses the freezing point of PDB (K_f = 7.1^{o}C/molal) by 3.5^{o}C. If you dissolve the same solid in H_2O (K_f = 1.86^{o}C/molal) to the same final molality as you had it in the PDB, what would the change in freezing point be?
3. You are instructed to add about 2 g of the unknown solid when determining the freezing point of the solution. Suppose you accidentally add 5 g of solid. Will the measured freezing point of the solution be higher or lower as a result of this mistake? Explain.
4. Cleaning PDB from the test tube after the experiment could be a long and arduous process. Describe the procedure that you will use to clean PDB (and the unknown solid) from your test tube after your experiments are complete.

4.R: Determination of the Molar Mass by Freezing Point Depression (Lab Report)

Lab Report: Determination of Molar Mass by Freezing Point Depression

Experimental Data

Unknown ID number: ______________________

Part 1: Freezing Point of Pure PDB	
Mass of large empty test tube	
Mass of test tube & PDB	
Mass of PDB (by difference)	

Part 2: Freezing Point of PDB-Unknown Mixture (~2 g)	
Mass of vial & unknown	
Mass of vial & unknown minus Sample I	
Mass of unknown added (by difference)	

Part 3: Freezing Point of PDB-Unknown Mixture (~4 g)	
Mass of vial & unknown	
Mass of vial & unknown minus Sample II	
Mass of unknown added (by difference)	
Total mass of unknown added	

Temperature Measurements: Record the temperature every 30 seconds as the pure solvent and two solutions are cooled. Note the temperature at which any solid first appears.

Time Elapsed (minutes)	Temperature (°C)		
---	Pure Solvent (PDB only)	Solution I (PDB + Sample I)	Solution II (PDB + Sample II)
0			
0.5			
1.5			
2			
2.5			
3			
3.5			
4			
4.5			
5			
5.5			
6			
6.5			
7			

Time Elapsed (minutes)	Temperature (°C)		
7.5			
8			
8.5			
9			
9.5			
10			
10.5			
11			
11.5			
12			
12.5			
13			
13.5			

Graphical Analysis of Data

Use Excel to create three separate graphs of “Temperature versus Time” for the pure solvent and the two solutions studied. Each graph should have an appropriate title and labeled axes with an appropriate scale. Add two trendlines to the data points of each graph. You can do this by hand with a ruler or by using Excel. The first line is applied to data points that correspond to the cooling of the liquid stat: these are the points on the steep part of the graph. The second line is applied to data points that correspond to the co-existence of both the solid and liquid (freezing): these are the points on the part of the graph where the temperature levels out. Extrapolate the two trendlines towards each other until they intersect. The temperature at the point of intersection is the solvent freezing point and should be clearly shown on each graph. **Attach your three graphs to this report.**

Record the freezing point temperatures obtained from the graphs below:

- Pure PDB ________________°C
- Solution I _______________________°C
- Solution II ______________________°C

Calculation of Molar Mass

Complete the table below with the results of your calculations. Be sure to include all units. Note that K_f PDB = 7.10 °C·kg·mol^{-1}.

	Solution I	Solution II
Mass of PDB added		
Total mass of unknown added		
Freezing point of pure PDB		
Freezing point of solution		
Total Freezing point depression, ΔT_f		
Molality of solution		
Moles of unknown in solution		
Molar Mass of unknown		

- Unknown number ______________________ has an average molar mass of _______________________g/mol.

- On a separate sheet of paper, show the equations used and calculations performed for molality of the solutions, moles of unknown in the solutions, and molar mass of the unknown. **Attach your sheet to this report.**

Questions

1. Look up the freezing point of para-dichlorobenzene. This may be found in the *CRC Handbook of Chemistry and Physics* (library) or from various online sources.

- Freezing Point of PDB =__________________________
- Reference source used:

2. Using the freezing point from your reference source, determine the percentage error in your experimentally measured freezing point. Show your calculation below, and be sure to report your answer to the correct number of significant figures.

3. Suppose you find out that the actual molar mass of your unknown solid is exactly three times larger than the value you determined experimentally. What could you conclude about the nature of your unknown solid and the assumptions you made in your calculations in such a case? Explain.

5: Le Chatelier's Principle (Experiment)

Objectives

- To perturb chemical reactions at equilibrium and observe how they respond.
- To explain these observations using Le Chatelier’s Principle.
- To relate Le Chatelier’s Principle to the concept of coupled reactions.

All chemical reactions eventually reach a state in which the rate of the reaction in the forward direction is equal to the rate of the reaction in the reverse direction. When a reaction reaches this state, it is said to be at chemical equilibrium. The concentrations of reactants and products at equilibrium are constant as a function of time. Thus, for a homogeneous aqueous system of the form

$$a\mathrm{A}(aq) + b\mathrm{B}(aq) \rightleftharpoons c\mathrm{C}(aq) + d\mathrm{D}(aq) \tag{5.1}$$

we can express the equilibrium-constant expression for this reaction as,

$$K_c = \frac{[\mathrm{C}]^c[\mathrm{D}]^d}{[\mathrm{A}]^a[\mathrm{B}]^b} \tag{5.2}$$

where the values of $[\mathrm{A}]$, $[\mathrm{B}]$, $[\mathrm{C}]$, and $[\mathrm{D}]$ correspond to the equilibrium concentrations (or equilibrium positions) of all the aqueous chemical components, and a, b, c, and d are their respective stoichiometric coefficients. Note that for a heterogeneous system including pure solids or liquids of the form

$$a\mathrm{A}(aq) + b\mathrm{B}(s) \rightleftharpoons c\mathrm{C}(aq) + d\mathrm{D}(l) \tag{5.3}$$

the pure liquids and solids do not appear in the equilibrium-constant expression:

$$K_c = \frac{[\mathrm{C}]^c}{[\mathrm{A}]^a} \tag{5.4}$$

It has been observed that when a reaction at equilibrium is perturbed by applying a stress, the reaction will respond by shifting its equilibrium position so as to counteract the effect of the perturbation/stress. In other words, the concentrations of the reactants and products will shift so that the relationship described by Equation 5.2 is again satisfied. This idea was first proposed by Henri-Louis Le Chatelier and has since been referred to as “Le Chatelier’s Principle.”

Note that when a reaction makes more products as a response to the perturbation, we call it a right-shift. When a reaction makes more reactants in response to the perturbation, we call it a left-shift. We often designate these respective shifts by drawing right and left arrows below the chemical equation.

For chemical reactions at equilibrium in aqueous solutions, the most common types of perturbations include changing the concentration of one of the aqueous solutes, changing the concentrations of all aqueous solutes by changing the total solution volume, or changing the temperature. The general responses of an aqueous system to these particular perturbations are tabulated below.

Perturbation	Effect on Equilibrium Position	Effect on K_c
Increase in concentration of a single reactant, or, decrease in concentration of a single product.	Shift to the right	None
Decrease in concentration of a single reactant, or, increase in concentration of a single product.	Shift to the left	None
Decrease in all aqueous concentrations due to an increase in solution volume resulting from the addition of solvent	Shift towards the side with more solute particles	None
Increase in all aqueous concentrations due to a decrease in solution volume resulting from the removal of solvent (evaporation)	Shift towards the side with fewer solute particles	None

Perturbation	Effect on Equilibrium Position	Effect on K_c
Increase temperature of an exothermic reaction	Shift to the left	Decrease
Decrease temperature of an exothermic reaction	Shift to the right	Increase
Increase temperature of an endothermic reaction	Shift to the right	Increase
Decrease temperature of an endothermic reaction	Shift to the left	Decrease
Addition of an inert substance, catalyst, pure liquid, or pure solid	None	None

Notice that only a temperature change can affect the value of K_c; in all other cases, the value of K_c remains constant.

In this experiment, you will perturb reactions that have attained equilibrium. You will then observe how each reaction responds to that perturbation in order to restore equilibrium. In your report, you describe these changes in terms of Le Chatelier's Principle.

Part A – Acid-Base Equilibrium

Here you will use **coupled equilibria** to change the equilibrium position of an acid-base reaction. In order to understand how coupled equilibria work consider the reactions described by the chemical equations below:

$$\mathrm{A}(aq) \rightleftharpoons \mathrm{B}(aq) \tag{5.5}$$

$$\mathrm{B}(aq) + \mathrm{C}(aq) \rightleftharpoons \mathrm{D}(aq) \tag{5.6}$$

Notice that the species $\mathrm{B}(aq)$ is common to both reactions. The presence of this common species couples these two reactions.

We can perturb the equilibrium position of Reaction 5.6 by the addition of some $\mathrm{C}(aq)$.The addition of $\mathrm{C}(aq)$ will cause the equilibrium position of Reaction 5.6 to shift right in accordance with Le Chatelier's Principle. This right shift in the equilibrium position of Reaction 5.6 will also result a corresponding decrease in the concentration of $\mathrm{B}(aq)$. Because $\mathrm{B}(aq)$ is also present in Reaction 5.5, the decrease in the concentration of $\mathrm{B}(aq)$ will in turn result in a right shift in the equilibrium position of Reaction 5.5. Thus, the addition of $\mathrm{C}(aq)$ to Reaction 5.6 actually results in a right shift in the equilibrium position of Reaction 5.5 because the equilibria are coupled.

In Part A we will observe the effect of various solutes on an acid-base indicator (a weak acid) at equilibrium. The equilibrium system can be written in the general form

$$\mathrm{HA(aq)} \rightleftharpoons \mathrm{H^+(aq)} + \mathrm{A^-(aq)} \tag{5.7}$$

The equilibrium-constant expression for this reaction is

$$K_a = \frac{[\mathrm{H^+}][\mathrm{A^-}]}{[\mathrm{HA}]} \tag{5.8}$$

where we denote the equilibrium constant, K, with a subscript a for acid. In this experiment, HA and $\mathrm{A^-}$ are the acidic and basic forms of the indicator bromothymol blue. Since the two forms are different colors, you will be able to determine which form is predominant in the equilibrium mixture. In other words, you will be able to determine whether the equilibrium position lies to the left (more reactants and less products) or whether the equilibrium lies to the right (more products and less reactants).

Your goal will be to find a reagent that will shift the position of this equilibrium to the opposite side, and then another reagent that will shift it back towards its original position. Instead of directly adding HA or $\mathrm{A^-}$ to the system, you will affect these shifts by adding $\mathrm{H^+}$ or $\mathrm{OH^-}$. Note that in order to determine the effect of $\mathrm{OH^-}$ we must consider a second chemical reaction that shares a common species with the Reaction 5.7. The second reaction is the autoionization of water, which can be described by the equation

$$\mathrm{H_2O(l)} \rightleftharpoons \mathrm{H^+(aq)} + \mathrm{OH^-(aq)} \tag{5.9}$$

The equilibrium constant for this reaction is denoted by K_w, where the subscript w stands for water, and the associated equilibrium constant expression is

$$K_w = [\mathrm{H}^+][\mathrm{OH}^-] \tag{5.10}$$

Because Reactions 5.7 and 5.9 share a common chemical species (H^+), you can use the concept of coupled equilibria to shift the equilibrium position of Reaction 5.7 by increasing or decreasing the concentration of $\mathrm{OH}^-(\mathrm{aq})$.

Part B – Solubility Equilibrium

Here you will test the effects of changing temperature and volume on the solubility of a slightly soluble salt at equilibrium. Some examples of slightly soluble salts are AgCl, $\mathrm{Cu(OH)_2}$, $\mathrm{PbCl_2}$, and $\mathrm{Fe_2S_3}$ and are general referred to as being “insoluble in water.” However, a very small amount of each of these substances dissolves in aqueous solutions, but the amount is so small that we often classify each of these compounds as “insoluble.”

This type of equilibrium is often called a **solubility equilibrium** because it is written in the direction of the dissolution of the solid, as shown in the following example:

$$\mathrm{A_xB_y}(s) \rightleftharpoons x\mathrm{A}^+(aq) + y\mathrm{B}^-(aq) \tag{5.11}$$

The equilibrium-constant expression for Reaction 5.11 is

$$K_{sp} = [\mathrm{A}^+]^x[\mathrm{B}^-]^y \tag{5.12}$$

where we denote the equilibrium constant, K, with a subscript *sp* for **solubility product**.

Now let’s consider the process of precipitation. In a typical precipitation reaction two aqueous salt solutions are mixed together resulting in the production of an insoluble salt. Notice that this process corresponds to a left shift of Reaction 5.11, and so Equation 5.12 can also be used to examine the conditions required for the precipitation of a solid to occur. We can denote the product $[\mathrm{A}^+]^x[\mathrm{B}^-]^y$ under arbitrary conditions (not necessarily at equilibrium) as,

$$Q_{sp} = [\mathrm{A}^+]^x[\mathrm{B}^-]^y \tag{5.13}$$

where Q_{sp} is called the solubility product **reaction quotient**. Note that, upon mixing two solutions, one containing A^+ and the other containing B^-, if $Q_{sp} < K_{sp}$ the system is not at equilibrium, but since no solid $\mathrm{A_xB_y}$ is present the reaction cannot shift to the right and therefore no reaction will be observed. In contrast, if $Q_{sp} > K_{sp}$ the solution contains an excess of aqueous species, and Reaction 5.11 will shift left, forming the solid precipitate $\mathrm{A_xB_y}$ until the system reaches a state of equilibrium where $Q_{sp} = K_{sp}$. Thus, we can use the values of Q_{sp} and K_{sp} to predict the conditions under which a precipitation reaction will occur.

In Part B we will study the solubility equilibrium of $\mathrm{PbCl_2(s)}$. We will observe the effect on this solubility equilibrium of changes in solution volume (quantity of solvent) and temperature. We will express these changes in terms of the respective values of Q_{sp} and K_{sp}.

Part C – Complex Ion Equilibrium

Certain metal ions, most often transition metals, exist in solution as complex ions in combination with other ions or molecules, called ligands. Common ligands include $\mathrm{H_2O}$, $\mathrm{NH_3}$, Cl^-, and OH^-. Many of these complex ions exhibit vibrant colors in solution. For example, the $\mathrm{Co(H_2O)_6^{2+}(aq)}$ complex ion is pink and the $\mathrm{CoCl_4^{2-}(aq)}$ complex ion is blue.

In Part C you will study the following complex ion formation reaction:

$$\mathrm{Co(H_2O)_6^{2+}(aq) + 4\,Cl^-(aq) \rightleftharpoons CoCl_4^{2-}(aq) + 6\,H_2O(l)} \tag{5.14}$$

The equilibrium-constant expression for Reaction 5.14 is

$$K_f = \frac{[\mathrm{CoCl_4^{2-}}]}{[\mathrm{Co(H_2O)_6^{2+}}][\mathrm{Cl^-}]^4} \tag{5.15}$$

where we denote the equilibrium constant, K, with a subscript f for complex ion *formation*.

Your goal in Part C is to observe how Reaction 5.14 shifts from its equilibrium position as the result of various perturbations.

Part D – Dissolving Insoluble Solids

In Part D you will use coupled equilibria to affect the solubility equilibrium of $\mathrm{Zn(OH)_2(s)}$. The solubility equilibrium can be described by the equation

$$\mathrm{Zn(OH)_2(s) \rightleftharpoons Zn^{2+}(aq) + 2\,OH^-(aq)} \qquad K_{sp} = 5\times10^{-17} M^3 \tag{5.16}$$

Notice that $K_{sp} << 1$ for this reaction, demonstrating that $\mathrm{Zn(OH)_2(s)}$ is only very slightly soluble in aqueous solution.

Now consider the reactions described by the following chemical equations, each of which shares a common species with the Reaction 5.16:

$$\mathrm{H_2O(l) \rightleftharpoons H^+(aq) + OH^-(aq)} \qquad K_w = 1\times10^{-14} M^2 \tag{5.17}$$

$$\mathrm{Zn^{2+}(aq) + 4\,OH^-(aq) \rightleftharpoons Zn(OH)_4^{2-}(aq)} \qquad K_f = 3\times10^{15} M^{-4} \tag{5.18}$$

$$\mathrm{Zn^{2+}(aq) + 4\,NH_3(aq) \rightleftharpoons Zn(NH_3)_4^{2+}(aq)} \qquad K_f = 1\times10^{9} M^{-4} \tag{5.19}$$

Because Reactions 5.17, 5.18, and 5.19 each share a common species with Reaction 5.16 they can be coupled together. In Part D of this experiment, you will observe the effect of coupling each of these equilibria on the solubility of $\mathrm{Zn(OH)_2(s)}$.

Procedure

Equipment

From lab/locker: Six large test tubes, test tube rack, stirring rod, scoopula, small 10-mL graduated cylinder, large 100-mL graduated cylinder, 400-mL beaker, hot plate

From hallway: Ice Machine

Chemicals

Bromothymol blue, a 6 M strong acid, a 6 M strong base, 0.3 M $\mathrm{Pb(NO_3)_2}$ (*aq*), 0.3 M HCl (*aq*), $\mathrm{CoCl_2 \cdot 6\,H_2O}$ (*s*), 12 M HCl (*aq*), 0.1 M $\mathrm{Zn(NO_3)_2}$ (*aq*), 0.1 M $\mathrm{Mg(NO_3)_2}$ (*aq*), 6 M NaOH (*aq*), 6 M HCl (*aq*), 6 M $\mathrm{NH_3}$ (*aq*) (often labeled as $\mathrm{NH_4OH}$), and deionized water

Safety And Waste Disposal

- 12-M HCl, is extremely caustic and great care must be taken to avoid contact with eyes or skin. The bottle should be kept in a plastic tray and not removed from the fume hood. Should any of this solution enter your eyes rinse immediately in the emergency eyewash. Should any of this solution come in contact with your skin rinse with copious amounts of water and apply saturated sodium bicarbonate to the affected area from the stock bottle located on the sink.
- The solutions you will use in Part B contain lead. Be certain that all of these lead-containing solutions are disposed of in the proper waste container and rinse your hands following this procedure.
- Many of the chemicals used in this lab are hazardous to the environment. All waste must be disposed of in the hazardous-waste container in the fume hood. Rinse all glassware directly into the waste container twice using a small squirt bottle to be certain all hazardous waste ends up in the waste container.

General Procedural Notes

The amounts of reagents used in this experiment are approximate only. If you are unsure how to estimate a milliliter, then measure out about one milliliter of water using your graduated cylinder, transfer this amount to a large test tube, and then use this approximate volume as a reference throughout the experiment.

All glassware needs to be rinsed at least once with deionized water. It is not necessary to dry glassware since all reagent volumes are approximate and all solutions are aqueous.

Part A: Acid-Base Equilibrium

Here you will find a reagent that will shift the acid-base equilibrium given by Reaction 5.7 in one direction and then a second reagent that will cause the equilibrium position to shift back in the opposite direction.

Reagents needed for this part are: deionized water, bromothymol blue solution, a 6 M strong acid, and a 6 M strong base.

1. Add approximately 5 mL of deionized water to a large test tube. Add 3 drops of the bromothymol blue indicator solution. Report the color of your solution on your datasheet.
2. Your solution from Step 1 currently contains one form of bromothymol blue (see background). Now predict which of the two 6 M reagents you obtained, the strong acid or the strong base, will cause a color change in your solution by making the

bromothymol blue indicator shift to its other form. Add the 6 M reagent of your choice drop-by-drop and if your solution changes color, write the color of the solution and formula of the reagent on your datasheet. If the addition of your reagent does not result in a color change, try other reagents until you are successful.

3. Since equilibrium systems are reversible, it is possible to shift a reaction left or right repeatedly by changing the conditions. Now find another 6 M reagent that will cause your solution from Step 2 to revert back to its original color. Add the 6 M reagent of your choice drop-by-drop and if your solution changes color, write the formula of the reagent on the datasheet. If the addition of your second reagent does not result in a color change, try other reagents until you are successful.

Part B: Solubility Equilibrium

You will test the effects of changing the solution volume and temperature on the solubility equilibrium of lead (II) chloride.

Reagents needed are: 0.3 M $\mathrm{Pb(NO_3)_2}$ (*aq*), 0.3 M HCl (*aq*), and deionized water.

1. Set up a hot-water bath by filling a 400-mL beaker about half-full with tap water and heating the water using a hot plate while you work on the following steps. The hot-water bath will be used in Step 6 (and also in Part C).
2. Measure 5.0 mL of the 0.3 M $\mathrm{Pb(NO_3)_2}$ solution into a large test tube using a 10-mL graduated cylinder.
3. Thoroughly rinse the graduated cylinder with deionized water, then measure 5.0 mL of the 0.3 M HCl solution using the 10-mL graduated cylinder.
4. Add about 1 mL of the 0.3 M HCl solution to the $\mathrm{Pb(NO_3)_2}$ solution in the large test tube. Stir the mixture gently using your stirring-rod and record your observations on your data sheet.
5. Continue to add the 0.3 M HCl solution to the $\mathrm{Pb(NO_3)_2}$ solution in the large test tube in roughly 1 mL increments until you just begin to see white $\mathrm{PbCl_2}$ solid appear in your test tube. To confirm that the solid is present, let the test tube sit on the bench for about 3 minutes, allowing all solid to settle to the bottom where it is easier to see. On your data sheet record, the total volume of 0.3 M HCl needed to produce the solid.
6. Put the test tube containing the solid into your hot-water bath from Step 1. Stir the contents of the test tube gently for a few seconds using the glass stirring-rod and record your observations on your datasheet. Continue heating and stirring until a change is observed.
7. Make a cold-water bath by filling a 400-mL beaker half full with tap water and ice. Cool the test tube down by placing it into this cold-water bath. Observe what happens and record your observations. Save this cold-water bath for use in Part C.
8. Dispose of the remainder of the 0.3 M HCl solution in your 10-mL graduated cylinder by pouring it into the appropriate waste container and then rinse the cylinder using deionized water. Next measure 5.0 mL of deionized water using this graduated cylinder.
9. Add about 1 mL of deionized water to the large test tube containing the $\mathrm{PbCl_2}$ solid. Stir the mixture and observe what happens. Continue to add the deionized water in 1 mL increments until the white $\mathrm{PbCl_2}$ solid *just dissolves* (or disappears). On your datasheet, record the volume of water needed to dissolve the solid.
10. Pour the contents of the large test tube into your large graduated cylinder and measure the total volume of the solution. Record this volume on your datasheet.
11. Dispose of all solutions used in Part B in the proper waste container. Rinse all glassware twice using a wash bottle of deionized water to ensure that all of the lead solution has been removed and transferred to the waste container. Save the hot- and cold-water baths for Part C.

Part C: Complex Ion Equilibrium

Here you will test the effects of changing the volume and temperature on the complex ion equilibrium between Co(H2O)62+ (aq) and CoCl42- (aq) as in Reaction 5.14.

Reagents needed for this part are: $\mathrm{CoCl_2 \cdot 6\,H_2O}$ (*s*), 12 M HCl (*aq*) (*do not remove from fume hood*), and deionized water.

1. Reheat your hot-water bath from part B to a near boil. The hot-water bath will be used in Step 4.
2. Put a few small crystals (an amount that fits at the tip of the scoopula) of $\mathrm{CoCl_2 \cdot 6\,H_2O}$ (*s*) in a large test tube. Working in the fume hood, carefully add 2 mL of 12 M HCl (*aq*) (*this acid is extremely caustic; see cautions in the safety section of this experiment*). Add a 1/2 inch magnetic stir bar and stir to dissolve the crystals. Record the color of this solution.
3. Using a 10-mL graduated cylinder, add deionized water to the solution in your test tube in approximately 2 mL increments, stirring after each addition, until no further color change occurs. Record the new color.
4. Place the test tube into the hot-water bath from Step 1 and record any color change.
5. Cool the solution down in the cold-water bath and record any color change.

Part D: Dissolving Insoluble Solids

Here you will further examine how one reaction can affect the behavior of another reaction when the reactions share one or more common chemical species.

Reagents needed for are: 0.1 M $Zn(NO_3)_2$ (*aq*), 0.1 M $Mg(NO_3)_2$ (*aq*), 6 M $NaOH$ (*aq*), 6 M HCl (*aq*), and 6 M NH_3 (*aq*) (often labeled as NH_4OH).

1. Label three large test tubes A, B, and C. Add about 2 mL of 0.1 M $Zn(NO_3)_2$ solution to each test tube. Add one drop of 6 M $NaOH$ solution to each test tube. Stir each solution and record your observations.
2. Into test tube A, add at least 20 drops of 6 M HCl (*aq*) drop-by-drop while stirring. Record all observations.
3. Into test tube B, add at least 20 drops of 6 M $NaOH$ (*aq*) drop-by-drop while stirring. Record all observations.
4. Into test tube C, add at least 20 drops of 6 M NH_3 (*aq*) drop-by-drop while stirring. Record all observations.
5. Label three additional large test tubes D, E, and F. Add about 2 mL of 0.1 M $Mg(NO_3)_2$ solution to each test tube. Add one drop of 6 M $NaOH$ solution to each test tube. Stir each solution and record your observations.
6. Into test tube D, add at least 20 drops of 6 M HCl (*aq*) drop-by-drop while stirring. Record all observations.
7. Into test tube E, add at least 20 drops of 6 M $NaOH$ (*aq*) drop-by-drop while stirring. Record all observations.
8. Into test tube F, add at least 20 drops of 6 M NH_3 (*aq*) drop-by-drop while stirring. Record all observations.

5.P: Le Chatelier's Principle (Pre-Lab)

1. Cadmium hydroxide, an important component of NiCd batteries, is only slightly soluble in water.

- Write a reaction for the dissolution of cadmium hydroxide in water.
- Write the K_{sp} expression for cadmium hydroxide.
- You add a small amount of NaOH to a test tube containing cadmium nitrate solution. Initially nothing happens, but as you add more NaOH you begin to observe the formation of a white, powdery solid that resembles snowflakes. Eventually, all the precipitate settles to the bottom of your test tube. Explain why you do not see the precipitate initially as you first begin to add NaOH (aq).
- Explain why you eventually do see a precipitate.
- Which of the following two reagents: $\mathrm{HNO_3}$ or KOH, do you think will increase the solubility of cadmium hydroxide solution?Explain your answer.

2. The following complex ion formation equilibrium takes place in water:

$$\mathrm{Cu(CN)_4^{2-}(aq) + 4\,NH_3(aq) \rightleftharpoons Cu(NH_3)_4^{2+}(aq) + 4\,CN^-(aq)} \tag{5.P.1}$$

- Write the K_f expression for this complex ion reaction
- The color of $\mathrm{Cu(CN)_4^{2-}}$ (aq) is light blue, while the color of $\mathrm{Cu(NH_3)_4^{2+}}$ is violet. Suppose you mix solutions of $\mathrm{Cu(NO_3)_2}$, NaCN and $\mathrm{NH_3}$ in a test tube and allow the reaction to reach equilibrium. The color of the solution is light blue at this point. Given that the above reaction is endothermic, should you heat the solution, or cool the solution in order to change the color from light blue to violet? Explain your answer using Le Chatelier’s principle.

3. Congo Red (CR) is a pH indicator which has a blue color in its acidic form and red color in its basic form. When dissolved in water, CR forms a red-colored solution.

- Write a reaction for the equilibrium between the two forms of congo red. Use HCR for the acidic form and CR^- for the basic form.
- You make a solution of CR in water. Which of the following reagents, 6 M HCl or 6 M NaOH, will cause the solution to change color? Explain your answer using the concept of coupled reaction.

5.R: Le Chatelier's Principle (Lab Report)

Name: ______________________________ Lab Partner: __________________________

Date: __________________________ Lab Section: ___________________

Part A – Equilibrium and an Acid-Base Indicator

Equilibrium system:

$$\mathrm{HA(aq)} \rightleftharpoons \mathrm{H^+(aq)} + \mathrm{A^-(aq)} \tag{5.R.1}$$

Observations

Record your results upon completing each of the following steps:

Step 1: Color of bromothymol blue in distilled water	
Step 2: Name of reagent "A" causing color change when added	
Step 3: Name of reagent "B" causing a return to original color	

Analysis

- Complete the following:

The acidic form of the bromothymol blue indicator, HA (*aq*), is _____________ in color.
The basic form of the bromothymol blue indicator, $\mathrm{A^-}$ (*aq*), is _____________ in color.

- Explain why reagent A (in Step 2) caused the color change observed.
- Explain why reagent B (in Step 3) caused the color change observed.

Part B – Solubility Equilibrium and K_{sp}

Equilibrium system:

$$\mathrm{PbCl_2(s)} \rightleftharpoons \mathrm{Pb^{2+}(aq)} + 2\,\mathrm{Cl^-(aq)} \tag{5.R.2}$$

Observations

Step 4: Observations upon addition of just 1.0 mL of HCl to the $\mathrm{Pb(NO_3)_3}$ solution	
Step 5: Total volume of HCl required for noticeable precipitation	mL
Step 6: Observations upon placing the test tube with precipitate in hot water	
Step 7: Observations upon placing the test tube with precipitate in cold water	
Step 9: Volume of water added to just dissolve $\mathrm{PbCl_2}$ precipitate	mL
Step 10: Total solution volume upon completion	mL

Analysis

- Why didn't any solid $\mathrm{PbCl_2}$ form immediately upon addition of 1 mL of HCl (*aq*) in Step 4? What condition must be met by $[\mathrm{Pb^{2+}}]$ and $[\mathrm{Cl^-}]$ if solid $\mathrm{PbCl_2}$ is to form?
- Consider your observation in hot water in Step 6:

  In which direction did the equilibrium shift? ___________________

Did the value of K_{sp} get smaller or larger? ____________________

Is the dissolution of $\mathrm{PbCl_2}$ (*s*) exothermic or endothermic? ____________________

Explain below.

- Explain why the solid $\mathrm{PbCl_2}$ dissolved when water was added to it in Step 9. What was the effect of this water on $[\mathrm{Pb}^{2+}]$, $[\mathrm{Cl}^-]$, and Q_{sp}? In which direction would such a change drive the equilibrium system?
- The point at which the $\mathrm{PbCl_2}$ precipitate just dissolves in Step 9 can be used to determine the value of K_{sp} for this equilibrium system, where $K_{sp} = [\mathrm{Pb}^{2+}][\mathrm{Cl}^-]^2$. Calculate $[\mathrm{Pb}^{2+}]$ and $[\mathrm{Cl}^-]$ in the final solution (consider the "dilution effect"). Then use these equilibrium concentrations to determine the value of K_{sp} for this system. Show all work below.

Part C – Complex Ion Equilibria

Equilibrium system:

$$\underbrace{\mathrm{Co(H_2O)_6^{2+}(aq)}}_{\text{Pink}} + 4\,\mathrm{Cl^-(aq)} \rightleftharpoons \underbrace{\mathrm{CoCl_4^{2-}(aq)}}_{\text{Blue}} + 6\,\mathrm{H_2O(l)} \tag{5.R.3}$$

Observations

Step 2: Color of solution in 12 M HCl	
Step 3: Color of solution upon addition of water	
Step 4: Color of solution in hot water	
Step 5: Color of solution in cold water	

Analysis

- What form of the complex ion, $\mathrm{Co(H_2O)_6^{2+}}$ (*aq*) or $\mathrm{CoCl_4^{2-}}$ (*aq*), is predominate in:

  The 12 M HCl (*aq*) ___________________

  The diluted solution ___________________

  The heated solution ___________________

- Explain why you obtained the observed color in 12 M HCl (*aq*) (Step 2).
- Explain the observed color change that occurred when water was added to the solution in Step 3. Consider how water affects the ion concentrations and Q in this system.
- Consider your observations in the hot water bath in Step 4.

  In which direction did the equilibrium shift? _______________________

  Did the value of K get smaller or larger? _______________________

  Is the reaction (as written) exothermic or endothermic? _______________________

  Explain.

Part D – Dissolving Insoluble Solids

Equilibrium system:

$$\mathrm{Zn(OH)_2(s)} \rightleftharpoons \mathrm{Zn^{2+}(aq)} + 2\,\mathrm{OH^-(aq)} \quad K_{sp} << 1$$

Observations

Step 1: Adding 1 drop of NaOH (*aq*) to $\mathrm{Zn(NO_3)_2}$ (*aq*)	
Step 2: Tube A: Effect when HCl (*aq*) is added	

Step 3: Tube B: Effect when $NaOH(aq)$ is added	
Step 4: Tube C: Effect when NH_3 (*aq*) is added	

Analysis

- Explain your observation upon addition of HCl (*aq*) to the precipitate in Tube A. You must consider the various equilibria that are occurring in solution and the effect of HCl on $[OH^-]$.
- Explain your observations upon addition of $NaOH$ (*aq*) to the precipitate in Tube B. Consider the various equilibria that are occurring in solution and remember that Zn^{2+} forms stable complex ions with OH^- at sufficiently high concentrations.
- Explain your observations upon addition of NH_3 (*aq*) to the precipitate in Tube C. Consider the various equilibria that are occurring in solution and remember that Zn^{2+} forms stable complex ions with NH_3.

Part E

Equilibrium system:

$$\mathrm{Mg(OH)_2(s) \rightleftharpoons Mg^{2+}(aq) + 2\,OH^-(aq)} \quad K_{sp} \ll 1 \tag{5.R.4}$$

Observations

Step 1 Adding 1 drop of $NaOH(aq)$ to $Mg(NO_3)_2$ (*aq*)	
Step 2: Tube A: Effect when HCl (*aq*) is added	
Step 3: Tube B: Effect when $NaOH(aq)$ is added	
Step 4: Tube C: Effect when NH_3 (*aq*) is added	

Analysis

- Based on your observations in Steps 3 and 4 do you think that Mg^{2+} forms stable complex ions? Explain your reasoning.

6: Determination of Kc for a Complex Ion Formation (Experiment)

Objectives

- Find the value of the equilibrium constant for the formation of FeSCN^{2+} by using the visible light absorption of the complex ion.
- Confirm the stoichiometry of the reaction.

In the study of chemical reactions, chemistry students first study reactions that go to completion. Inherent in these familiar problems—such as calculation of theoretical yield, limiting reactant, and percent yield—is the assumption that the reaction can consume *all* of one or more reactants to produce products. In fact, most reactions do not behave this way. Instead, reactions reach a state where, after mixing the reactants, a stable mixture of reactants and products is produced. This mixture is called the *equilibrium state*; at this point, a chemical reaction occurs in both directions at equal rates. Therefore, once the equilibrium state has been reached, no further change occurs in the concentrations of reactants and products.

The equilibrium constant, K, is used to quantify the equilibrium state. The expression for the equilibrium constant for a reaction is determined by examining the balanced chemical equation. For a reaction involving aqueous reactants and products, the equilibrium constant is expressed as a ratio between reactant and product concentrations, where each term is raised to the power of its reaction coefficient (Equation 6.2). When an equilibrium constant is expressed in terms of molar concentrations, the equilibrium constant is referred to as K_c. The value of this constant at equilibrium is always the same, regardless of the initial reaction concentrations. At a given temperature, whether the reactants are mixed in their exact stoichiometric ratios or one reactant is initially present in large excess, the ratio described by the equilibrium constant expression will be achieved once the reaction composition stops changing.

$$a\mathrm{A}(aq) + b\mathrm{B}(aq) \rightleftharpoons c\mathrm{C}(aq) + d\mathrm{D}(aq) \tag{6.1}$$

with

$$K_c = \frac{[\mathrm{C}]^c[\mathrm{D}]^d}{[\mathrm{A}]^a[\mathrm{B}]^b} \tag{6.2}$$

We will be studying the reaction that forms the reddish-orange iron (III) thiocyanate complex ion, $\mathrm{Fe(H_2O)_5SCN^{2+}}$ (Equation 6.3). The actual reaction involves the displacement of a water ligand by thiocyanate ligand, $\mathrm{SCN^-}$ and is often call a ligand exchange reaction.

$$\mathrm{Fe(H_2O)_6^{3+}(aq) + SCN^-(aq) \rightleftharpoons Fe(H_2O)_5SCN^{2+}(aq) + H_2O(l)} \tag{6.3}$$

For simplicity, and because water ligands do not change the net charge of the species, water can be omitted from the formulas of $\mathrm{Fe(H_2O)_6^{3+}}$ and $\mathrm{Fe(H_2O)_5SCN^{2+}}$; thus $\mathrm{Fe(H_2O)_6^{3+}}$ is usually written as $\mathrm{Fe^{3+}}$ and $\mathrm{Fe(H_2O)_5SCN^{2+}}$ is written as $\mathrm{FeSCN^{2+}}$ (Equation 6.4). Also, because the concentration of liquid water is essentially unchanged in an aqueous solution, we can write a simpler expression for K_c that expresses the equilibrium condition only in terms of species with variable concentrations.

$$\mathrm{Fe^{3+}(aq) + SCN^-(aq) \rightleftharpoons FeSCN^{2+}(aq)} \tag{6.4}$$

In this experiment, students will create several different aqueous mixtures of $\mathrm{Fe^{3+}}$ and $\mathrm{SCN^-}$. Since this reaction reaches equilibrium nearly instantly, these mixtures turn reddish-orange very quickly due to the formation of the product $\mathrm{FeSCN^{2+}}$ (*aq*). The intensity of the color of the mixtures is proportional to the concentration of product formed at equilibrium. As long as all mixtures are measured at the same temperature, the ratio described in Equation 6.4 will be the same.

Measurement of $[\mathrm{FeSCN^{2+}}]$

Since the complex ion product is the only strongly colored species in the system, its concentration can be determined by measuring the intensity of the orange color in equilibrium systems of these ions. Two methods (visual inspection and spectrophotometry) can be employed to measure the equilibrium molar concentration of $\mathrm{FeSCN^{2+}}$, as described in the *Procedure* section. Your instructor will tell you which method to use. Both methods rely on Beer's Law (Equation 6.5). The absorbance, A, is directly proportional to two parameters: c (the compound's molar concentration) and path length, l (the length of the sample through which the light travels). Molar absorptivity ε, is a constant that expresses the absorbing ability of a chemical species at a certain wavelength. The absorbance, A, is roughly correlated with the color intensity observed visually; the more intense the color, the larger the absorbance.

$$A = \varepsilon \times l \times c \tag{6.5}$$

In the visual inspection method, you will match the color of two solutions with different concentrations of FeSCN^{2+} by changing the depth of the solutions in a vial. As pictured in Figure 6.1, the solution on the left is more concentrated than the one on the right. However, when viewed from directly above, their colors can be made to "match" by decreasing the depth of the more concentrated solution. When the colors appear the same from above, the absorbances, A, for each solution are the same; however, their concentrations and path lengths are not.

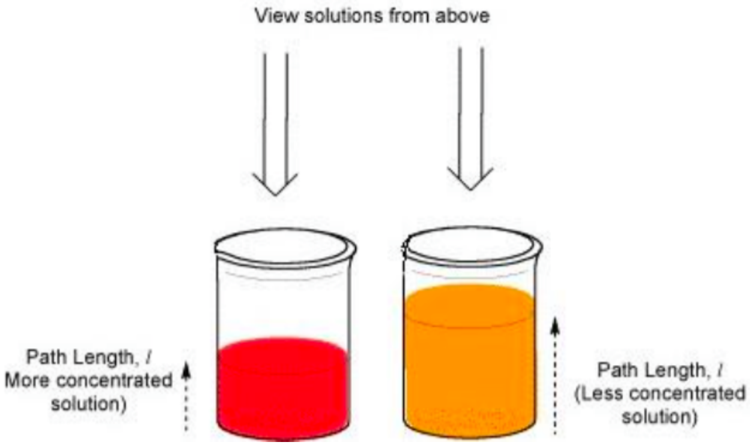

Figure 6.1: Solution depths (path lengths) of two samples containing different concentrations of FeSCN^{2+} can be adjusted so that their apparent color looks the same when viewed from above.

If a standard sample with a known concentration of FeSCN^{2+} is created, measurement of the two path lengths for the "matched" solutions allows for the calculation of the molar concentration of FeSCN^{2+} in any mixture containing that species (Equation 6.6). This equation is an application of Beer's Law, where $\frac{A}{\varepsilon}$ for matched samples are equal. Therefore, the product of the path length, l, and molar concentration, c, for each sample are equal.

$$l_1 \times c_1 = l_2 \times c_2 \tag{6.6}$$

By using a spectrophotometer, the absorbance, A, of a solution can also be measured directly. Solutions containing FeSCN^{2+} are placed into the spectrophotometer, and their absorbances at 447 nm are measured. In this method, the path length, l, is the same for all measurements. Rearrangement of Equation 6.5 allows for the calculation of the molar concentration, c, from the known value of the constant $\varepsilon \times l$. The value of this constant is determined by plotting the absorbances, A, vs. molar concentrations, c, for several solutions with known concentration of FeSCN^{2+} (Figure 6.2). The slope of this calibration curve is then used to find unknown concentrations of FeSCN^{2+} from their measured absorbances.

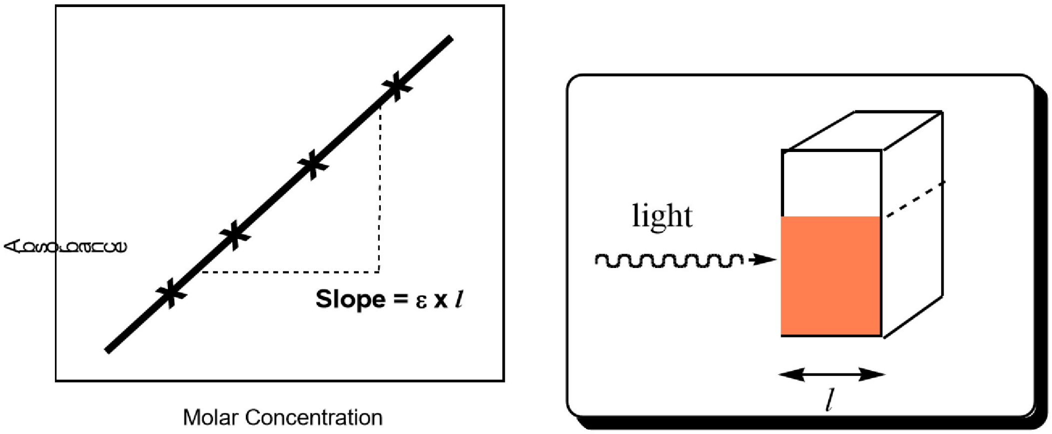

Figure 6.2: Plots of $Absorbance$ vs. $Concentration$ for solutions with known $[\text{FeSCN}^{2+}]$ can be used as a calibration curve. This plot is used to determine $[\text{FeSCN}^{2+}]$ in solutions where that value is not known. The path length, l, is demonstrated in the diagram of a cuvet.

Calculations

In order to determine the value of K_c, the equilibrium values of $[\text{Fe}^{3+}]$, $[\text{SCN}^{-}]$, and $[\text{FeSCN}^{2+}]$ must be known. The equilibrium value of $[\text{FeSCN}^{2+}]$ was determined by one of the two methods described previously; its initial value was zero, since no FeSCN^{2+} was added to the solution.

The equilibrium values of $[\text{Fe}^{3+}]$ and $[\text{SCN}^{-}]$ can be determined from a reaction table ('ICE' table) as shown in Table 6.1. The initial concentrations of the reactants—that is, $[\text{Fe}^{3+}]$ and $[\text{SCN}^{-}]$ prior to any reaction—can be found by a dilution calculation based on the values from Table 6.2 found in the procedure. Once the reaction reaches equilibrium, we assume that the reaction has shifted forward by an amount, x. The equilibrium concentrations of the reactants, Fe^{3+} and SCN^{-}, are found by subtracting the equilibrium $[\text{FeSCN}^{2+}]$ from the initial values. Once all the equilibrium values are known, they can be applied to Equation 6.4 to determine the value of K_c.

Table 6.1: ICE TABLE

Reaction 6.4	Fe^{3+}	$+\ \text{SCN}^{-}$	$\rightleftharpoons \text{FeSCN}^{2+}$
Initial Concentration	$[\text{Fe}^{3+}]_i$	$[+\text{SCN}^{-}]_i$	0
Change in Concentration	$-x$	$-x$	$+x$
Equilibrium Concentration	$[\text{Fe}^{3+}]_i - x$	$[+\text{SCN}^{-}]_i - x$	$x = [\text{FeSCN}^{2+}]$

The reaction "ICE" table demonstrates the method used in order to find the equilibrium concentrations of each species. The values that come directly from the experimental procedure are found in the shaded regions. From these values, the remainder of the table can be completed.

Standard Solutions of FeSCN^{2+}

In order to find the equilibrium $[\text{FeSCN}^{2+}]$, both methods require the preparation of standard solutions with known $[\text{FeSCN}^{2+}]$. These are prepared by mixing a small amount of dilute $ceKSCN$ solution with a more concentrated solution of $ceFe(NO3)3$. The solution has an overwhelming excess of Fe^{3+}, driving the equilibrium position far towards products. As a result, the equilibrium $[\text{Fe}^{3+}]$ is very high due to its large excess, and therefore the equilibrium $[\text{SCN}^{-}]$ must be very small. In other words, we can assume that ~100% of the SCN^{-} is reacted, meaning that SCN^{-} is a limiting reactant resulting in the production of an equal amount of FeSCN^{2+} product. Examine the Kc-expression to prove this to yourself. In summary, due to the large excess of Fe^{3+}, the equilibrium concentration of FeSCN^{2+} can be approximated as the initial concentration of SCN^{-}.

Procedure

Materials and Equipment

Solutions: Iron(III) nitrate (2.00 x 10^{-3} M) in 1 M HNO_3; Iron(III) nitrate (0.200 M) in 1 M HNO_3; Potassium thiocyanate (2.00 x 10^{-3} M).

Materials:

50-mL beaker x 2, Test tubes, stirring rod, 1-mL and 2-mL volumetric pipet, 5-mL and 10-mL volumetric pipet*, 10- mL graduated cylinder, Pasteur pipets, spectrometers, and cuvets* (2).

*must obtain from stockroom

Safety

The iron(III) nitrate solutions contain nitric acid. Avoid contact with skin and eyes; wash hands frequently during the lab and wash hands and all glassware thoroughly after the experiment. Collect all your solutions during the lab and dispose of them in the proper waste container.

Part A: Solution Preparation

Label two clean, dry 50-mL beakers, and pour 30 mL of 2.00 x 10^{-3} M $Fe(NO_3)_3$ (already dissolved by the stockroom in 1 M HNO_3) into one beaker. Then pour 20 mL of 2.00 x 10^{-3} M KSCN into the other beaker. At your work area, label five clean and dry medium test tubes to be used for the five test mixtures you will make. Using your volumetric pipet, add 5.00 mL of your 2.00 x 10^{-3} M $Fe(NO_3)_3$ solution into each of the five test tubes. Next, using your 1-mL and 2-mL volumetric pipet, add the correct amount of KSCN solution to each of the labeled test tubes, according to the table below. Rinse out your volumetric pipet with deionized water, and then use it to add the appropriate amount of deionized water into each of the labeled test tubes. Stir each solution thoroughly with your stirring rod until a uniform orange color is obtained. To avoid contaminating the solutions, rinse and dry your stirring rod after stirring each solution.

Table 6.2: Test Mixtures

Mixture	$Fe(NO_3)_3$ Solution	KSCN Solution	Water
1	5.00 mL	5.00 mL	0.00 mL
2	5.00 mL	4.00 mL	1.00 mL
3	5.00 mL	3.00 mL	2.00 mL
4	5.00 mL	2.00 mL	3.00 mL
5	5.00 mL	1.00 mL	4.00 mL

Five solutions will be prepared from 2.00 x 10^{-3} M KSCN and 2.00 x 10^{-3} M $Fe(NO_3)_3$ according to this table. Note that the total volume for each mixture is 10.00 mL, assuming volumes are additive. If the mixtures are prepared properly, the solutions will gradually become lighter in color from the first to the fifth mixture. Use this table to perform dilution calculations to find the initial reactant concentrations to use in Figure 6.3.

Part B:

1. Preparation of a Standard Solution of $FeSCN^{2+}$

Before examining the five test mixtures, prepare a standard solution with a known concentration of $FeSCN^{2+}$. Obtain 15 mL of **0.200 M** $Fe(NO_3)_3$. (Note the different concentrations of this solution.) Rinse your volumetric pipet with a few mL of this solution, and add 10.00 mL of this solution into a clean and dry large test tube. Using your graduated cylinder, add 8.00 mL deionized water to the large test tube. Rinse your volumetric pipet with a few mL of 2.00 x 10^{-3} M KSCN. Then add 2.00 mL of the KSCN solution to the large test tube. Mix the solution with a clean and dry stirring rod until a uniform dark-orange solution is obtained. This solution should be darker than any of the other five solutions prepared previously.

2. Spectrophotometric Determination of $[\mathrm{FeSCN}^{2+}]$

Your instructor may ask you to measure the absorbance of each test mixture using a spectrophotometer rather than by matching the colors with a standard solution. In this case, you will be creating a calibration curve that plots the absorbance of FeSCN^{2+} at 447 nm vs. molar concentration. From this curve, you can determine $[\mathrm{FeSCN}^{2+}]$ in each mixture from the absorbance at 447 nm.

A few dilutions of the standard solution prepared in Part B1 will be used to prepare four standard solutions for your calibration curve. In addition, one 'blank' solution containing only $\mathrm{Fe(NO_3)_3}$ will be used to zero the spectrophotometer. The instructor will decide if each group of students will work alone or with other groups to prepare the standards. Table 6.3 shows the preparation method for each of the standards.

Table 6.3: Standard Solutions for Calibration Curve

Tube	Composition
Blank	0.200 M $\mathrm{Fe(NO_3)_3}$ in 1 M $\mathrm{HNO_3}$ (**Stock Solution Part B**)
1	**Standard Solution** (Part B)
2	4.00 mL **Standard Solution** + 1.00 mL $\mathrm{H_2O}$
3	4.00 mL **Standard Solution** + 2.00 mL $\mathrm{H_2O}$
4	4.00 mL **Standard Solution** + 3.00 mL $\mathrm{H_2O}$

Four standard solutions and one blank solution will be prepared for the calibration curve. Your instructor will tell you if you need to create all these solutions or if groups can share solutions.

Fill a cuvet with the blank solution and carefully wipe off the outside with a tissue. Insert the cuvet and make sure it is oriented correctly by aligning the mark on the cuvet towards the front. Close the lid. Your instructor will show you how to zero the spectrophotometer with this solution. Set aside this solution for later disposal. For each standard solution in Table 6.3, rinse your cuvet three times with a small amount (~0.5 mL) of the standard solution to be measured; dispose of the rinse solution each time. Then fill the cuvet with the standard, insert the cuvet as before and record the absorbance reading. Finally, repeat this same procedure with five mixtures with unknown $[\mathrm{FeSCN}^{2+}]$ prepared in Part A, Table 6.2 . The $[\mathrm{FeSCN}^{2+}]$ in this solutions will be read from the calibration curve.

6.P: Determination of Kc for a Complex Ion Formation (Pre-Lab)

A student mixes 5.0 mL of 0.00200 M $Fe(NO_3)_3$ with 5.0 mL 0.00200 M KSCN. She finds that the concentration of $FeSCN^{2+}$ in the equilibrium mixture is 0.000125 M. Follow these steps to determine the corresponding experimental value of K_c for the reaction of Fe^{3+} and SCN^- to produce this complex ion. Show your calculations for each step below and then place the appropriate value(s) in the equilibrium (or 'ICE') table near the bottom of the page.

- *Step 1.* Calculate the molarity of Fe^{3+}, SCN^-, and $FeSCN^{2+}$ initially present after mixing the two solutions, but prior to any reaction taking place. ($M_1V_1 = M_2V_2$)
- *Step 2.* Determine the expression and initial value for Q_c. Then give the appropriate signs of the concentration changes for each species in terms of the reaction's shift, or x, into the 'ICE' table.
- *Step 3.* Fill in the equilibrium value for the molarity of $FeSCN^{2+}$. From this, you can determine the value of x.
- *Step 4.* Given the value of x, determine the equilibrium molarities of Fe^{3+} and SCN^-.

'ICE' Table

	$Fe^{3+}(aq)$	$+ \quad SCN^-(aq)$	$\rightleftharpoons \quad FeSCN^{2+}(aq)$
I			
C			
E			

- *Step 5.* Give the correct expression for K_c for this equation. Then calculate the value of K_c for the reaction from the equilibrium concentrations. Use correct significant figures.
- *Step 6.* On the reverse side, complete an 'ICE' table using this same procedure, but using a different reaction stoichiometry: $Fe^{3+} + 2\,SCN^- \rightleftharpoons Fe(SCN)_2^{2+}$ (ignore that fact that this is incorrect since the reaction is not balanced with respect to charge). Assume that the equilibrium concentration of $FeSCN^{2+}$ is 0.0000625 M, or one-half its previous value. Remember how the reaction stoichiometry affects the expression for K_c.

6.R: Determination of Kc for a Complex Ion Formation (Lab Report)

Name: ______________________________ Lab Partner: __________________________

Date: __________________________ Lab Section: ___________________

Part A: Initial concentrations of Fe^{3+} and SCN^- in Unknown Mixtures

Experimental Data

Tube	**Reagent Volumes (mL)**			**Initial Concentrations (M)**	
--	2.00 x 10^{-3} M $Fe(NO_3)_3$	2.00 x 10^{-3} M KSCN	Water	Fe^{3+}	SCN^-
1	5.00	5.00	0.00		
2	5.00	4.00	1.00		
3	5.00	3.00	2.00		
4	5.00	2.00	3.00		
5	5.00	1.00	4.00		

- Show a sample dilution calculation for $[Fe^{3+}]$ initial in Tube #1 only

Part B and C: The Standard $FeSCN^{2+}$ Solution (Visual Method)

Given that 10.00 mL of **0.200 M** $Fe(NO_3)_3$, 2.00 mL of 0.00200 M KSCN, and 8.00 mL of water

- Equilibrium $[FeSCN^{2+}]$ in Standard Solution: _____________ M

> Note that since $[Fe^{3+}] \gg [SCN^-]$ in the Standard Solution, the reaction is forced to completion, thus causing all the SCN^- to convert to $FeSCN^{2+}$.

- Show the stoichiometry and dilution calculations used to obtain this value.

Equilibrium Concentrations of $FeSCN^{2+}$ in Mixtures:

Tube	**Solution Depths (mm)**		$[FeSCN^{2+}]_{equil}$ **(M)**
--	Mixtures	Standard	--
1			
2			
3			
4			
5			

- Show a sample calculation for $[FeSCN^{2+}]_{equil}$ in Tube #1 only.

Part D: Spectrophotometric Method

Calibration Curve Data

Tube	**Absorbance**	$[FeSCN^{2+}]$ **(M)**
Blank		
1		

Tube	Absorbance	$[\mathrm{FeSCN}^{2+}]$ (M)
2		
3		
4		

- Show a sample dilution calculation for $[\mathrm{FeSCN}^{2+}]$ in Tube #1 and 2 only.

> Note that since $[\mathrm{Fe}^{3+}] \gg [\mathrm{SCN}^-]$ in the Standard Solution, the reaction is forced to completion, thus causing all the added SCN^- to be converted to FeSCN^{2+}.

- Plot Absorbance vs.$[\mathrm{FeSCN}^{2+}]$ for the standard solutions. Obtain an equation for the line. This can be used to determine the $[\mathrm{FeSCN}^{2+}]$ in the table below. Attach your plot to this report.

Test mixtures

Mixture	Absorbance	$[\mathrm{FeSCN}^{2+}]$ (M)
1		
2		
3		
4		
5		

- Show a sample calculation for $[\mathrm{FeSCN}^{2+}]$ in mixture 1.
- Linear Equation (A vs. $[\mathrm{FeSCN}^{2+}]$) of Calibration Curve: ____________________

Calculations and Analysis

The reaction that is assumed to occur in this experiment is:

$$\mathrm{Fe}^{3+}(\mathrm{aq}) + \mathrm{SCN}^-(\mathrm{aq}) \rightleftharpoons \mathrm{FeSCN}^{2+}(\mathrm{aq}) \tag{6.R.1}$$

- Write the equilibrium constant expression for the reaction.
- Create a ***Reaction Table*** (or ICE table), as in Table 1, to demonstrate how the values below are calculated. Use the data for Mixture #1 only. Start with the known values for the initial concentrations of each species and the final value of $[\mathrm{FeSCN}^{2+}]$ from the data table on the previous page. Show how you find the value of the stoichiometric change in reaction concentrations that occurs, and the resulting equilibrium concentrations of the reactants.
- Show a sample calculation for the value of K_c using the data for Tube #1.

Using the same method you outlined above, complete the table for all the equilibrium concentrations and value of K_c:

Tube	Equilibrium Concentrations (M)			K_c
--	Fe^{3+}	SCN^-	FeSCN^{2+}	--
1				
2				
3				
4				

- Average value of K_c _________________ (Use reasonable number of significant digits, based on the distribution of your K_c values.)

Optional Analysis: Is an alternative reaction stoichiometry supported?

Suppose that instead of forming $\mathrm{FeSCN^{2+}}$, the reaction between $\mathrm{Fe^{3+}}$ and $\mathrm{SCN^-}$ resulted in the formation of $\mathrm{Fe(SCN)_2^+}$. The reaction analogous to Equation 6.R.1 would be:

$$\mathrm{Fe^{3+}(aq) + 2\,SCN^-(aq) \rightleftharpoons Fe(SCN)_2^+(aq)} \tag{6.R.2}$$

i.e., two moles of $\mathrm{SCN^-}$ displace two moles of $\mathrm{H_2O}$ in $\mathrm{Fe(H_2O)_6^{2+}}$ to make $\mathrm{Fe(SCN)_2(H_2O)_4^+}$.

- Write the equilibrium expression for this reaction.
- Create a ***Reaction Table*** for Mixture #1 only (or ICE table), as in Table 1, to clearly show how all the values below were obtained. Then show the calculation for the value of K_c for Tube 1. Pay special attention to the stoichiometry in this system. Begin by assuming that the equilibrium value for $[\mathrm{Fe(SCN)_2^+}]$ is be equal to $\frac{1}{2}[\mathrm{FeSCN^{+2}}]$ at equilibrium obtained previously. (This is because the moles of $\mathrm{SCN^-}$ assumed to be equal to the moles of complex ion product, $\mathrm{FeSCN^{2+}}$, in our standard solutions. In the alternate reaction, moles of product, $\mathrm{Fe(SCN)_2^+}$, equal ½ the number of moles of $\mathrm{SCN^-}$ added in the standard solutions.)

Using the method you outlined above, complete the table for all the equilibrium concentrations and values of K_c

Tube	Equilibrium Concentrations (M)			K_c
--	$\mathrm{Fe^{3+}}$	$\mathrm{SCN^-}$	$\mathrm{Fe(SCN)_2+}$	--
1				
2				
3				
4				
5				

- Average value of K_c ________________ (Use a reasonable number of significant digits, based on the distribution of your K_c values.)
- Based on the calculated values of K_c for each reaction stoichiometry, which reaction is the valid one?
- Briefly explain your conclusion. Compare the percent difference between the average value and the individual measurements. What does this tell you about the two possible stoichiometries? Do these reactions give consistent values of K_c for different initial reaction conditions?

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