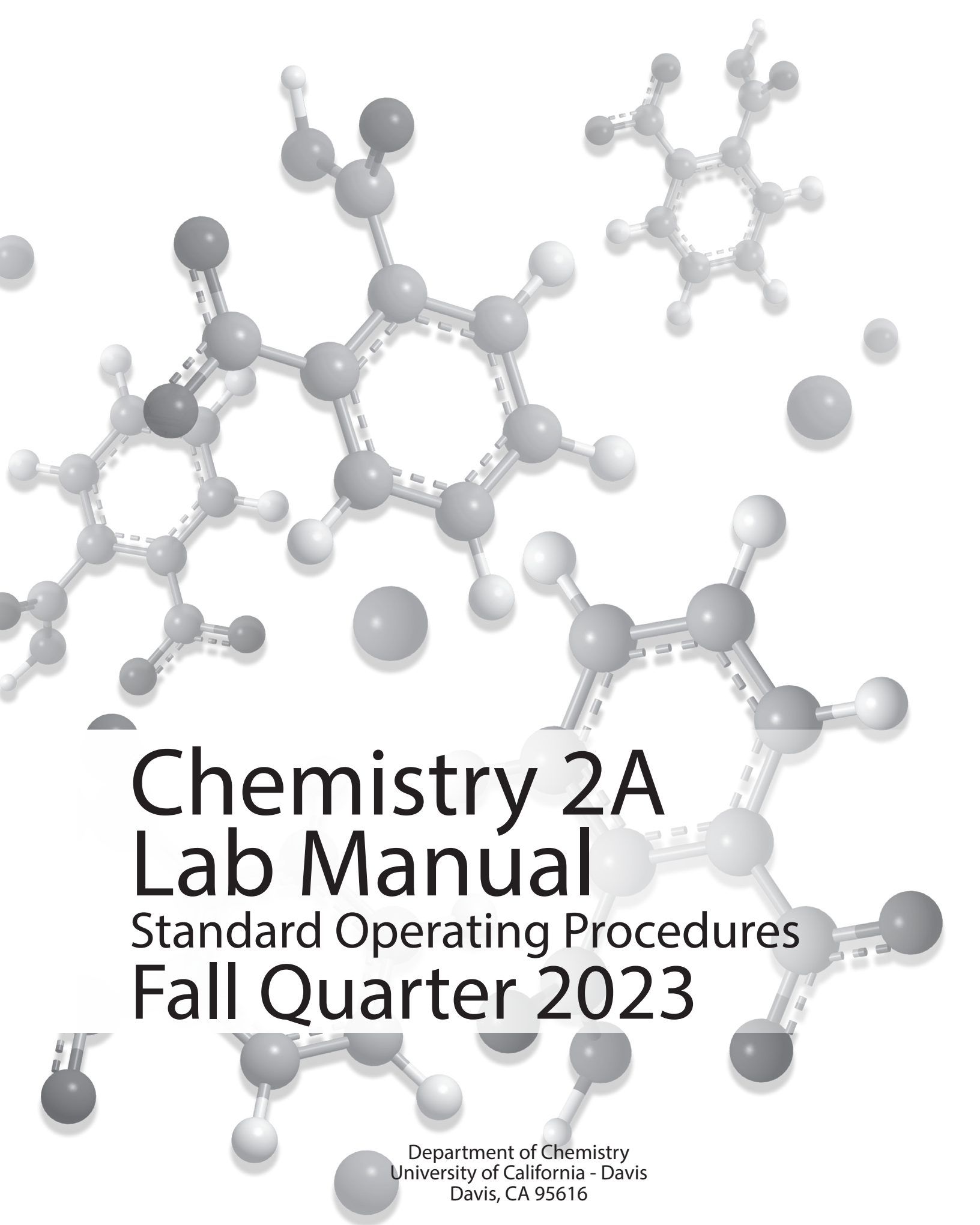


Chemistry 2A Lab Manual

Standard Operating Procedures

Fall Quarter 2023

Department of Chemistry
University of California - Davis
Davis, CA 95616

Student Name _____ **Locker #** _____

Laboratory Information

Teaching Assistant's Name _____

Laboratory Section Number _____

Laboratory Room Number _____

Dispensary Room Number **1060 Esau Hall**

Location of Safety Equipment Nearest to Your Laboratory

Safety Shower _____

Eye Wash Fountain _____

Fire Extinguisher _____

Fire Alarm _____

Safety Chemicals _____

Revision Date: August 2023

Preface

Chemistry is an experimental science. Thus, it is important that students of chemistry do experiments in the laboratory to more fully understand that the theories they study in lecture and in their textbook are developed from the critical evaluation of experimental data. The laboratory can also aid the student in the study of the science by clearly illustrating the principles and concepts involved. Finally, laboratory experimentation allows students the opportunity to develop techniques and other manipulative skills that students of science must master.

The faculty of the Chemistry Department at UC Davis clearly understands the importance of laboratory work in the study of chemistry. The Department is committed to this component of your education and hopes that you will take full advantage of this opportunity to explore the science of chemistry.

A unique aspect of this laboratory program is that a concerted effort has been made to use environmentally less toxic or non-toxic materials in these experiments. This was not only done to protect students but also to lessen the impact of this program upon the environment. This commitment to the environment has presented an enormous challenge, as many traditional experiments could not be used due to the negative impact of the chemicals involved. Some experiments are completely environmentally safe and in these the products can be disposed of by placing solids in the wastebasket and solutions down the drain with copious amounts of water. Others contain a very limited amount of hazardous waste and in these cases the waste must be collected in the proper container for treatment and disposal. The Department is committed to the further development of ***environmentally safe experiments*** which still clearly illustrate the important principles and techniques.

The sequence of experiments in this Laboratory Manual is designed to follow the lecture curriculum. However, instructors will sometimes vary the order of material covered in lecture and thus certain experiments may come before the concepts illustrated are covered in lecture or after the material has been covered. Some instructors strongly feel that the lecture should lead the laboratory while other instructors just as strongly believe that the laboratory experiments should lead the lecture, and still a third group feel that they should be done concurrently. While there is no "best" way, it is important that you carefully prepare for each experiment by reading the related text material before coming to the laboratory. In this way you can maximize the laboratory experience.

Questions are presented throughout each experiment. It is important that you try to answer each question as it appears in the manual, as it will help you understand the experiment as you do it. In addition, you are encouraged to complete the report as soon after laboratory as possible, as this is much more efficient than waiting until the night before it is due.

In conclusion, we view this manual as one of continual modification and improvement. Over the past few years, many improvements have come from student comments and criticisms. We encourage you to discuss ideas for improvements or suggestions for new experiments with your TA. Finally, we hope you find this laboratory manual helpful in your study of chemistry.

Acknowledgments

This manual is the culmination of the efforts of many individuals.

Many faculty members have provided ideas for the creation of these laboratories and have made numerous suggestions regarding their implementation. Stockroom Dispensary Supervisors, both past and present, have had a role in helping to develop these experiments and, in particular, helping to ensure that the experiments are tailored to our laboratories here at UC Davis. Safety TAs, both past and present, have edited this manual to ensure that the experimental procedures are clear and current. In addition, many undergraduates have been involved in the development of experiments as part of undergraduate research projects.

Table of Contents

Introduction

Time Allocation and Grading

Below is an indication of the time allocation of each experiment. At the end of the quarter, the student's TA will sum the scores and give this to the instructor, who will modify it as described in the course syllabus.

Title of Experiment	Lab Periods Allocated
Introductory Laboratory Techniques	1
Online Nomenclature Test	N/A
Observing Chemical Reactions	1
Chemical Equilibrium	1
Strong Acid- Strong Base Titration	1
Acid Dissociation Constants and the Titration of a Weak Acid	1
Polyprotic Systems	1
Acid/Base Buffers	1
Solubility Product	1
On-Line Prelab Quizzes	N/A
Lab Notebooks - Pre-lab (eight)	N/A

On-Line Pre-laboratory Quizzes: Each 2 point pre-lab quiz must be completed **at least 1 hour** prior to attending the student's scheduled lab class. All three quiz questions must be answered correctly before the student will be allowed to perform the laboratory experiment. If the quiz is failed on the first attempt, the student has four more attempts to pass the quiz. Because the questions are chosen randomly, different questions may be generated on each attempt. Students who fail these quizzes are considered unprepared and unsafe to work in the laboratory and will not be allowed to begin the laboratory procedure until the TA is convinced the student is prepared. The TA will check the pre-laboratory write-up and quiz the student. The TA will allow entry into the laboratory only if the student answers the questions correctly and the pre-laboratory write-up is complete. This policy will be strictly enforced.

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. A number of 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. Accidents commonly occur when the following rules, as approved by the Chemistry Department Safety Committee, are not followed.

U.C. Davis Department of Chemistry Chem. 2 Series

Standard Operating Procedures

SAFETY RULES FOR TEACHING LABORATORIES

The following rules are designed for your safety in the laboratory. The Laboratory Instructor (LI = TA, Laboratory Supervisor, and/or Course Instructor) is required to enforce these rules and has the full backing of the Department of Chemistry Staff and Faculty. The LI is also required to enforce all laboratory experiment-specific safety procedures in carrying out the laboratory work. Violations of these rules will result in expulsion from the laboratory.

1. **No one is allowed in the laboratory without the supervision of a LI. No laboratory work will be done without supervision. Perform only authorized experiments, and only in the manner instructed. DO NOT alter experimental procedures, except as instructed.**

2. **Specific permission from your LI is required before you may work in any laboratory section other than the one to which you have been assigned.** Only laboratory rooms where the same laboratory course is operating may be used for this purpose.

3. **If you have a special health condition (asthma, pregnancy, etc.) or any personal health concerns, consult your medical professional before taking chemistry lab.**

4. **If you come to the laboratory with non-compliant goggles, shoes, or clothing, you will not be allowed to work in the laboratory. In that context, note** THERE ARE NO MAKE-UP LABORATORIES**.** Your course grade will be significantly lowered or you may fail the course if you do not meet the lab attire requirements.

5. **100% cotton lab coats are REQUIRED.**

6. **Approved safety goggles must be worn by all persons at all times.** At NO TIME are safety glasses of any kind acceptable in the laboratory. Safety goggles may not be modified in any manner.

7. **Clothing that completely covers the legs—including the skin between the top of the shoe and the bottom of the pant leg—must be worn at all times in the laboratory** (tights or leggings are NOT suitable leg covering). Inadequate protection often leads to injury. Avoid wearing expensive clothing to lab as it may get damaged.

8. **Closed-toe, closed-heel shoes that completely cover the entire foot must be worn at all times.**

9. **Confine long hair while in the laboratory.**

10. **Horseplay and carelessness are not permitted and are cause for expulsion from the laboratory. You are responsible for everyone's safety.**

11. **Absolutely NO food or drinks are to be stored or consumed in the laboratory.** Contact lenses and cosmetics (hand lotion, lip balm, etc.) are not to be applied and medications are not to be consumed while in the laboratory.

12. Skateboards, rollerblades, and other such personal equipment must be stored outside of the laboratory. Personal electronics are only permitted when needed for the laboratory. Because cell phones or other personal electronic media can easily be damaged or contaminated in the laboratory, use of such devices is at the student's own risk.

13. **Learn the location and how to operate the nearest eyewash fountain, safety shower, fire extinguisher, and fire alarm box.** Basic first aid for any chemical splash is to wash the affected area for at least 15 minutes and seek medical attention. Use the emergency shower if appropriate, removing contaminated clothing for thorough washing. If the safety shower or eyewash is activated, the exposed person should be accompanied to the Student Health Center for further evaluation.

14. **Laboratory doors must remain closed except when individuals are actively entering or exiting the lab.**

15. **The student must have at least** ONE UNGLOVED HAND **when outside the laboratory.** Gloves are presumed to be contaminated and must not come into contact with anything outside the laboratory except chemical containers. Only use the **ungloved hand** to open doors, hold on to stair rails, or push elevator buttons.

16. **All activities in which toxic gases or vapors are used or produced must be carried out in the fume hood.**

17. **Mouth suction must never be used to fill pipets.**

18. **Containers of chemicals may not be taken out of the laboratory except to the dispensary for refill/replacement or to exchange full waste jugs for empty ones.** All containers must be **closed with the appropriate cap** before you take them into the hallway to the dispensary. Always use a bottle carrier when transporting chemicals and waste.

19. **Put all hazardous waste into the appropriate waste container(s) provided in your laboratory.** Do not overfill waste containers.

20. **All incidents, near misses, injuries, explosions, or fires must be reported at once to the LI.** In case of serious injury or fire (even if the fire is out), the LI or Lab Supervisor must call 911. The student must always be accompanied to the Student Health Center.

21. **Keep your working area clean – immediately clean up ALL spills or broken glassware. Dispose sharps in the appropriate container. Do not dispose pipette tips in regular trash.** Clean off your lab workbench before leaving the laboratory.

You must sign the Safety Acknowledgement sheet before you may work in the lab. If you have questions about these rules and procedures, please ask your LI before starting any laboratory work in this course.

Experiments

Introductory Laboratory Techniques

Introduction

Welcome to the Chemistry 2 laboratory! Chemistry is an experimental science, and you will find that experimentation can help you better understand lecture material. In the laboratory you will go over many practical applications of theories you learn in class. Use the laboratory as a study aid to help you understand chemistry, and to have fun!

Many students do not enjoy laboratory and do not find it helpful because they take a "cookbook" approach to chemistry. That is, they are thinking, "I mix 1 gram of this with 5 mL of that to get a blue solution with white stuff at the bottom." They do nothing more than follow the "recipe" without thinking about what is happening in the test tube and how it relates to their studies, the world, or even existence as we know it. Since we do not let you eat the end results of what you cook in lab, if you take the cookbook approach, you are going to have a poor experience in the laboratory and an especially hard time completing your laboratory reports.

This lab manual is written to help you avoid such a bad experience and to help you develop skills in solving problems. You will not find recipes in your experiments; you are given considerable leeway in designing your own experiments. Whenever you need a lab technique, you will be given complete instructions on how to execute it, but you must be able to figure out how to apply those techniques in discovering the solutions to the problems presented. It is critical that you read the experiment before coming to the laboratory, and attempt to understand the theory behind the experiment and the methods you will use in the laboratory to investigate that theory.

Consider yourself an investigator while you are in the laboratory. For example, in a typical reaction, first find out "who done it"; what chemicals take part in the reaction? Then find out the culprits' "method"; is energy taken in or given off? Finally, you need to find out the consequences; what compound is formed? If you take this approach you will have a better laboratory experience, and you will have a much easier time writing the experimental report. In short, you will learn more and learn more easily.

This lab is designed to 1) acquaint you with the equipment in your locker, and 2) introduce you to some basic laboratory techniques. A word of warning: a few of you may find this and other beginning laboratories to be somewhat tedious, especially if you've had a good high school chemistry laboratory course. However, please be patient as the goal is to give all students a good common background so every student has an excellent chance of success with the later, more difficult experiments.

Remember that as pre-laboratory preparation, you should come to the laboratory with **Title, Purpose, Procedure, and Data Tables** written in your duplicating paper laboratory notebook. At the end of the laboratory period, you should have your TA sign and date your laboratory notebook near your data tables. You will turn in a completed post-lab report by your next lab period.

Common Laboratory Procedures

You will now do a set of simple experiments to learn the proper techniques for using the different equipment in the laboratory. You must read the common laboratory procedures section of this manual before beginning this part of the exercise. These pages describe the proper use of equipment.

A record of all data should be placed in your laboratory notebook. Also, all calculations should be clearly shown in the notebook. Finally, be sure to answer all questions before turning in your report to the teaching assistant.

Learning Goals

The following is a list of skills that you will use in this experiment.

Laboratory	• Using a balance • Handling lab glassware • Using a buret • Using a volumetric pipet • Using a Bunsen burner
Conceptual	• Precision and accuracy • Temperature dependence of the density of water
Data Analysis	• Calculation of density • Statistical analysis (average, standard deviation, 90% confidence limit • Calculation of mass percent • Percent Error

Procedure

Work in pairs on this experiment.

Each student must collect data and submit a separate report. The actual data analyses and the written reports must be done entirely independently of your lab partner or other students. Make sure that you avoid unauthorized collaboration and plagiarism. All suspected violations of the Code of Academic Conduct will be referred to Student Judicial Affairs.

Stock Chemicals Used

Chemical	Maximum Amount Used
Manganese Sulfate, monohydrate *(s)*	1.0-1.2 g

Part I. Measuring Volumes

A. Using a Pipet to Measure Volume

1. Draw about 400 mL of deionized water into a clean beaker, and let it stand for 15 minutes to equilibrate to room temperature. Note that there is only one deionized water tap at each sink in the lab room; make sure you use the correct tap.

2. Confirm that your 10.00 mL volumetric pipet is clean by filling to above the mark with deionized water and then letting it drain.

   Your pipet is a transfer pipet that is calibrated **"to deliver"** (TD) rather than **"to contain"** (TC). The last drop of liquid should not drain out of the tip of a TD pipet in normal use.

   However, there should be no water drops left on the side walls. The presence of such drops indicates that your pipet is dirty. Pipet cleaning solution is located in a 1 L bottle at the reagent counter. Follow the instructions on the label for cleaning. Remember that your pipet is calibrated to deliver.

3. Measure and record the mass of a clean 125 mL Erlenmeyer flask.

4. Measure and record the temperature of the room and the temperature of the water that was set aside in step 1. The two temperatures should agree before you continue. Read the thermometer to the closest one-tenth of a degree, using your best estimate. Please be especially careful with the thermometer.

5. Use your pipet to deliver 10.00 mL of the equilibrated water into the Erlenmeyer flask. Note the precision used here.

6. Measure and record the mass of the flask and the water.

Safety First

Wear your PPE.

Use a pipet bulb to fill your pipet. Never use another form of suction.

Hint

You will need to take turns with your locker-mate using the 10.00 mL volumetric pipet. One of you should start part B, using a buret to measure volume, while the other is doing part A.

Hint

You will use pipets in many of the experiments in Chemistry 2.

It is important that you always clean the pipet at the end of the lab, and rinse it thoroughly with deionized water before returning it to storage. Be sure to follow the instructions given in the Appendix for proper use of pipets.

7. Repeat steps 5 and 6 at least two additional times without emptying out your flask between trials.

B. Using a Buret to Measure Volume

8. Discard the water in your Erlenmeyer flask, and re-measure the mass of the flask. The inside of the flask does not need to be completely dry because any water left in it is from the previous procedure and is at the same temperature as the new water you will be adding.

9. Use a 25 mL buret and accurately measure out about 12 mL of room temperature deionized water from part A into the flask. You should read the buret to the closest one-hundredth mL (e.g., 12.14 mL). You will have to estimate the last digit.

   In your laboratory notebook, record your initial buret reading and your final buret reading. The volume of water delivered by the buret is the difference between the final and initial buret reading.

10. Measure and record the mass of the flask and the water.

11. Repeat steps 9 and 10 at least two additional times without emptying out your flask between trials.

Always clean your buret after use and rinse it with deionized water before storage. Furthermore, be sure you follow the instructions given in the Appendix for proper use of the buret.

C. Using a Beaker to Measure Volume

12. Measure and record the mass of a clean and dry 100 mL beaker. Note that this beaker needs to have a 50 mL graduation mark.

13. Use your clean and dry 100 mL beaker and carefully measure out 50 mL of your room temperature water.

14. Measure and record the mass of the beaker and the water.

15. Empty out your beaker and carefully measure out another 50 mL of your room temperature water. There is no need to reweigh the empty beaker.

16. Measure and record the mass of the beaker and water.

17. Repeat steps 15 and 16 at least two additional times.

Part II. Drying a Hydrate

1. As demonstrated by your TA, place a clean crucible on a wire triangle on an iron ring above a Bunsen burner. With the TA watching, light the Bunsen burner and adjust the flame and the iron ring so that the crucible is positioned in the hottest part of the flame.

2. Heat the crucible for 5 minutes to make sure it is dry, and then remove it from the wire triangle using crucible tongs and place it on your benchtop on top of a piece of wire gauze to cool.

3. After the crucible has returned to room temperature (approximately 5 minutes), measure and record its mass to one-thousandth of a gram (milligram).

4. Weigh into your crucible 1.0–1.2 g of manganese(II) sulfate monohydrate, $MnSO_4 \cdot H_2O$, recording the exact mass to one-thousandth of a gram (milligram).

5. Heat the crucible with its contents for 5 minutes, and then remove it to your benchtop on top of a piece of wire gauze using crucible tongs.

6. After the crucible and its contents have returned to room temperature, measure and record the mass.

7. Repeat steps 5 and 6 until the mass readings are consistent and the mass no longer decreases after heating.

8. Calculate the mass loss by your sample upon heating.

9. Transfer the contents of your crucible to the waste container located in the fume hood.

Clean-Up:

- Solid dry manganese(II) sulfate may be disposed of in the proper waste container found in the fume hood.
- Clean your volumetric pipet and buret with deionized water only. All other glassware may be cleaned with tap water and rinsed with deionized water.
    - You may also use pipet cleaning solution to clean your volumetric pipet if any drops cling to the sides after draining.
- Always let your glassware air-dry; **do not** attempt to dry your glassware with a paper towel as the towel may become lodged in the glassware.
    - If time permits, now would be a good time to also clean any other dirty glassware in your locker.
- Return the burets and pipets to their proper locations and place all of the glassware from your locker back in your locker.
- Clean your laboratory bench with your deionized water wash bottle and sponge. Once finished, ask your TA to sign your data sheets and lock your locker.

Data Analysis

Calculating the Density of Water:

An important skill learned in the CHE 2 series is coming to lab with tables prepared in your lab notebook for any data that you will collect during the experiment (i.e. masses of substances, volumes of solvents, temperatures, observations, etc.). An example of a data table is given below:

Data Type Collected (Units)	Data		
Temperature of water in beaker (°C)			
Mass of 125 mL Erlenmeyer flask (g)			
	Trial 1	**Trial 2**	**Trial 3**
Volume of DI water delivered using a volumetric pipet (mL)			
Mass of flask with water (g)			
Mass of water delivered (g)			

1. For each trial, calculate the mass of water delivered.

   For Part I.A, calculate the mass of water deivered by the volumetric pipet using the following formula.

   mass of water delivered (g) = mass of flask with water (g) - mass of flask (g)

2. For each trial, determine the volume of water delivered.

   For Part I. B, calculate the volume of water delivered by the buret using the following formula.

   volume of water delivered (mL) = final buret reading (mL) - initial buret reading (mL)

3. For each trial, calculate the density of water by dividing the mass of water delivered by the volume of water delivered:

$$Density\ (g/mL) = \frac{mass\ of\ water\ delivered\ (g)}{volume\ of\ water\ delivered\ (mL)}$$

4. Based on your 3 trials, what is the average density of water?

5. Calculate standard deviation for your average density of water. For help with any statistical analyses of your data, including calculating the average, standard deviation, and 90% confidence limit, please see the Statistical Treatment of Data section in the Appendix.

6. Calculate the 90% confidence limit for your average density of water.

7. Use the temperature of your water along with the values of mass and volume of water given in Table 1 to calculate the accepted values for the density of water.

8. Use the following formula and the accepted literature value for the density of water to calculate relative error.

$$Relative\ Error\ = \frac{|experimental\ result\ -\ accepted\ value|}{accepted\ value} \times 100\ \%$$

Table 1:

The volume occupied by 1.0000 g water weighed in air against stainless steel weights.

Temperature (°C)	Volume (mL)
18	1.0024
19	1.0026
20	1.0028
21	1.0030
22	1.0033
23	1.0035
24	1.0037
25	1.0040
26	1.0043

Table 1 gives the corrected volume in mL occupied by 1.0000 g of water when weighed in air against stainless steel weights for different temperatures. Two effects are included in this volume per 1.0000 g: first, the change in the density of water with temperature, and second, a much smaller correction due to buoyancy.

The buoyancy correction arises since the balance was set to zero with a certain mass of air on the balance pan. The volume of water displaces some of this air from the balance pan, and the displacement makes the water appear lighter than it really is. The contribution of buoyancy to the results in Table 1 is roughly 0.0011 mL per 1.0000 g of water.

9. Complete steps 1-8 to calculate the density of water when measured using a pipet, a buret, and a beaker.

Calculating Mass Percent of Water:

1. What was the mass, in grams, of your $MnSO_4 \cdot H_2O$ sample before heating?

2. What was the mass, in grams, of your $MnSO_4 \cdot H_2O$ sample after heating?

3. What was the mass, in grams, of water lost from your sample after heating?

4. Calculate the experimental mass percent of water in your $MnSO_4 \cdot H_2O$ sample. Use the following formula:

$$Mass\ Percent\ (\%)\ = \frac{Sample\ Mass_{initial} - Sample\ Mass_{final}}{Sample\ Mass_{initial}} \times 100\ \%$$

5. Calculate the theoretical mass percent of water by using the molecular weights for $MnSO_4 \cdot H_2O$ for the initial mass, and $MnSO_4$ for the final mass.
    - The molecular weights for $MnSO_4 \cdot H_2O$ and $MnSO_4$ are 169.0 g/mol and 151.0 g/mol respectively.
6. Use the formula for relative error, the experimental mass percent of water in your $MnSO_4 \cdot H_2O$ sample, and the theoretical mass percent of water to calculate relative error.

Observing Chemical Reactions

Introduction

An integral part of any experimental science is observing how the world behaves and drawing conclusions from the observed behavior. In this laboratory exercise you will mix chemicals and make observations about the resulting solutions. Your observations of these attempts will tell you whether or not the reactions actually occur, and from this data you will be able to plan a procedure for identifying and separating the salts in an unknown.

How do you know that mixing two chemicals results in a chemical reaction? Look for as many physical indications as possible. Does the color of the solution change? Does it heat up? Does it cool down? Is gas evolved? Use all of your senses except smell and taste; remember, never smell or eat any chemicals in the laboratory!

It cannot be emphasized enough that making good observations and writing them down, is critical to successful investigations in science. Think about how often you have said to yourself, "I'll remember the phone number until I get home," and then promptly forgotten it. It is much easier to forget something you have noted about a new chemical reaction, especially something you did not realize was significant at the time, than something you considered important in the first place. If you note a change or a lack of change, write it down!

The types of changes that you may observe in this lab can include color changes, bubbling, or precipitation reactions. Precipitation reactions are a particular type of reaction that results when an insoluble compound is formed from the mixing of two chemicals, and these types of reactions are very important in everyday life.

Is that mold in my kettle?

Water quality is one example in which precipitation chemistry is commonly used. Both surface and ground water may contain high levels of dissolved calcium and magnesium minerals, the primary cause of hard water. The higher the Ca^{2+} and Mg^{2+} content of water, the greater the degree of hardness. Aside from leaving unsightly stains in bathtubs, toilets, and sinks, hard water can leave mineral deposits, called "scale", in appliances and pipes, which reduces their efficiency and may cause pipes to burst. If you use a kettle to boil water, you may have noticed a white film of scale develop on the inside. Lime ($Ca(OH)_2$) and soda ash (Na_2CO_3) are often added to hard water in order to precipitate Ca^{2+} and Mg^{2+}. The insoluble precipitate settles at the bottom of the water tank and can be collected safely.

After determining which chemicals react in Part I of this lab, you will need to develop a scheme for the separation of a mixture of salts for Part II. Check with you TA to ensure that your scheme will work. Once you have an acceptable scheme, you will identify which two of the salts you have worked with are in your unknown solution.

Learning Goals

The following is a list of skills that you will use in this experiment.

Laboratory	• Recording detailed observations • Proper handling of acids/bases • Using a centrifuge • Decanting a solution
Conceptual	• Solubility rules • Double replacement reactions
Data Analysis	• Comparing and contrasting qualitative data (observation) • Developing experimental procedures from observations

Procedure

Work in pairs on this experiment.

Each student must collect data and submit a separate report. The actual data analyses and the written reports must be done entirely independently of your lab partner or other students. Make sure that you avoid unauthorized collaboration and plagiarism. All suspected violations of the Code of Academic Conduct will be referred to Student Judicial Affairs.

Stock Chemicals Used

Chemical	Maximum Amount Used
0.1 M Magnesium Nitrate (aq)	5 mL
0.1 M Strontium Nitrate (aq)	5 mL
0.1 M Aluminum Nitrate (aq)	5 mL
0.1 M Silver Nitrate (aq)	5 mL
6 M Hydrochloric Acid	<5 mL
6 M Nitric Acid	<5 mL
3 M Sulfuric Acid	<5 mL
6 M Sodium Hydroxide	<5 mL

Part I. Reactions of the Metal Salt Solutions

In this part of the experiment, you will experiment with four metal salts and four other reagents. Acquire approximately 5 mL of each metal salt solution in test tubes and bring them to your lab bench. The acids and bases will be found on the trays by your lab station.

Before Starting the Experiment:

- Please copy the observation table from the Data Analysis section into your lab notebook (be sure to make the table large).

React each reagent with the individual metal salts

1. Acquire approximately 5 mL of 6 M HCl in a test tube and bring it to your lab bench at the beginning.
2. Using a disposable transfer pipet, transfer approximately 1 mL of a 0.1 M metal salt solution to a clean test tube.
3. Using the dedicated transfer pipet attached to the reagent bottles, slowly add a couple of drops of one of the reagents to one of the metal salts. Record your observations.

Safety First

Use care when handling acids and bases.

Silver nitrate will stain clothing and skin, so always wear gloves.

Wear your goggles.

Recording Observations

Use care when recording your observations.

Be detailed, write them legibly in the table, and make sure you understand what you recorded!

Mix Slowly & Thoroughly

Take care in slowly and thoroughly mixing reagents to ensure that every possible reaction is observed within a homogeneous mixture. Failure to remove an ion can also lead to false positives.

4. Add a couple more drops and record your observations. Continue until you are sure that you have added an excess of the reagent. It will not take more than 0.5 mL to reach an excess of the reagents.

5. There are sixteen possible combinations of salts with reagents. Try them all and record any reactions you observe. You may also want to see what happens if you add more than one reagent to the salt solution.

As you work on this portion of the experiment, compare your results with your neighbors. If you seem to get different results, talk about why your results differ. Did one of you make an error, or are you just going about things differently?

Question A: In performing these reactions and any required dilutions, should you use tap water or deionized water? Why?

Question B: For each observed reaction between a reagent and a metal salt write a balanced chemical equation that shows what is occurring.

Clean-Up:

- Pour any solutions or solids containing silver or aluminum into the Cation Metal Waste jar in the fume hood.
- Pour the rest of the solutions into a 400 mL beaker for clean-up at the end of the laboratory.

Part II. Analyzing an Unknown

In this part of the experiment you will use the data you have accumulated to tell which of the salts are present in an unknown.

Your unknown contains two of the four metal salts you have worked with. Develop a procedure that will distinguish between these four compounds utilizing the table you have completed, and use it to identify the composition of your unknown. For help with this, see the Data Analysis section.

Hint

The centrifuge will also be evenly balanced if you place your test tube opposite another group's test tube containing a similar volume of unknown.

Be sure to label your test tube with graphite so you can identify it when the centrifuge stops!

You may need to be able to separate a solid precipitate from a solution. Instructions for separation follow.

1. Transfer the solution and the precipitate to a centrifuge test tube. Fill a second test tube with water until the volumes in the two test tubes are approximately the same.

2. Place the two test tubes in the centrifuge. The test tubes should be placed opposite each other so that their weight is balanced as the centrifuge spins.

As there are likely to be more people using the centrifuge, make sure that your test tubes are labeled so that you can identify them when the centrifuge stops.

3. Turn on the centrifuge and allow it to spin for a minute. When the centrifuge stops spinning, remove your test tube carefully—you do not want to disturb the solid at the bottom of the tube.

Complete removal of a salt from a solution

- If you are trying to completely remove a salt from a solution, add a little more of the reagent that caused the precipitation. If more solid forms in the solution, re-centrifuge and repeat this step.

4. Decant the supernatant solution from the solid and reserve. Avoid disturbing the precipitate when pouring off the solution.

5. Add a few milliliters of deionized water to the precipitate and stir. This washes any excess reagent away from the precipitate. Re-centrifuge. Decant the supernatant and combine with the reserved solution from Step 4.

You have now completed the separation of your precipitate from solution. Separate tests may be performed (as needed) on the precipitate or the supernatant solution.

Question C: Why do you test for complete precipitation if you are going to do any further chemical tests on the supernatant?

Clean-Up:

- Pour any solutions or solids containing silver or aluminum into the Cation Metal Waste container in the fume hood.
- Pour the rest of the solutions into your 400 mL beaker.
- Slowly add 3 grams of sodium bicarbonate to the solution in the beaker to neutralize the acid.
- Pour the neutralized solution in the sink with copious amounts of water.

Data Analysis

Observing Reactions of the Metal Salt Solutions

An important skill learned in the CHE 2 series is coming to lab with tables prepared in your lab notebook for any data that you will collect during the experiment (i.e. masses of substances, volumes of solvents, temperatures, observations, etc.).

Please copy the table below into your lab notebook. Make sure that each of the cells are large enough to write detailed observations during the experiment (feel free to take up a whole page in your lab notebook if needed).

Observation Table for Part I. Reaction of Metal Salt Solutions

Metal Ions	Reagents			
	HCl	NaOH	HNO_3	H_2SO_4
Ag^+				
Sr^{2+}				
Mg^{2+}				
Al^{3+}				

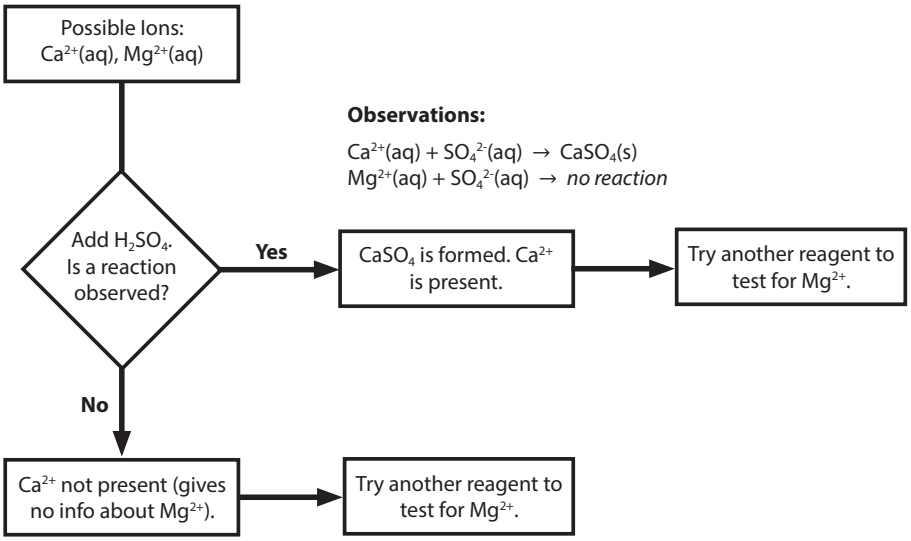
An example of a detailed observation may include some of the following information (be as descriptive as possible):

- Color change upon mixing of two reactants
- Formation of a precipitate (or insoluble solid)
- Formation of gas (seen as bubbling without heating the mixture)
- Temperature change (the vessel feels colder/warmer after reacting, indicating an endo- or exothermic reaction)

Analyzing an Unknown Metal Salt Solution

Use the observations collected in Part I to determine which 2 metal salts are present in your unknown. Trust your data, do not blindly follow solubility rules. In general, these tips may help:

- Using your observations, create a flowchart of which ions react with which reagents, similar to the one below.
- If multiple reactions are observed with the same reagent, compare your observations with Part I to identify the metals present in your unknown.



Ask Your TA

Some transition metals can have very interesting results. Talk to your TA if you want further information.

Solubility Rules

These rules are provided to help you understand the basic solubility rules of chemistry. These rules always have exceptions, as many things in chemistry do. As a consequence, remember to trust your observations.

1. Compounds that are soluble or mostly soluble:
    - Group 1, NH_4^+, chlorates, acetates, nitrates
    - Halides (except Pb^{2+}, Ag^+, and Hg_2^{2+})
    - Sulfates (except Ca^{2+}, Sr^{2+}, Ba^{2+}, Pb^{2+}, and Hg_2^{2+})
2. Compounds that are insoluble:
    - Hydroxides, sulfides (except above rules, and group 2 sulfides)
    - Carbonates, phosphates, chromates (except above rules)

Chemical Equilibrium

Introduction

Previously, you have only experienced reactions in lab that proceed to completion. An example of this type of reaction is the dissociation of HCl in water. Since hydrochloric acid is a strong acid, it completely dissociates in water. A 1.0 M HCl solution is essentially a solution of 1.0 M hydrogen ions and 1.0 M chloride ions in water. We can write this as:

$$HCl(aq) \rightarrow H^{+}(aq) + Cl^{-}(aq)$$

The same does not occur when the weak acid HF is dissolved in water. While a 1.0 M HF solution does contain hydrogen and fluoride ions, it also contains a significant quantity of undissociated hydrofluoric acid. We can write this as:

$$HF(aq) \rightleftharpoons H^{+}(aq) + F^{-}(aq)$$

The double arrow, as seen above, is used to indicate that the acid does not completely dissociate, and that the system has established chemical equilibrium. Equilibrium can be thought of as a balance between the reactants and products. Most reactions do not go to completion, but instead proceed to a point where both reactants and products are present. An important aspect of chemical equilibrium is that it can be established by starting with either reactants or products. The reaction will proceed in the forward or backward direction until coming to equilibrium, which depends on the concentration of reactants/products, temperature, and pressure.

In 1884, French chemist Henri Louis Le Châtelier presented his findings on the behavior of chemical systems at equilibrium. He found that when equilibrium was disturbed, by either changing concentration, pressure, or temperature, the reaction would re-establish equilibrium by proceeding in the direction that "relieved stress" on the system. This principle is generally referred to as Le Châtelier's Principle, and is as follows: when a stress is applied to a chemical system at equilibrium, the equilibrium shifts in a direction that reduces the effect of the stress.

Kidney Stones: a painful product of chemical equilibrium

Systems of equilibrium are extremely important for everyday life, particularly for living organisms. Many systems within organisms follow equilibrium dynamics, such as maintaining proper blood pH and oxygen levels, or the formation of kidney stones. Kidney stones can develop when the concentration of dissolved minerals and salts in urine is too high. In order to re-establish equilibrium, the minerals and salts will precipitate out of the urine and form solid, crystal masses. Kidney stones can be prevented with a special diet or by drinking plenty of water, both which keep the concentration of minerals and salts low enough that they won't precipitate.

In this qualitative experiment, you will observe different systems of equilibrium, and note the effect of added stress on each system. Make sure to record detailed observations and consider how the results relate to equilibrium topics you are studying in lecture.

Learning Goals

The following is a list of skills that you will use in this experiment.

Laboratory	• Use of indicators to interpret chemical systems (phenolphthalein and methyl orange) • Estimating volume with a disposable transfer pipet
Conceptual	• Le Châtelier's principle • Precipitation reactions in acidic or basic solutions
Data Analysis	• Interpretation of observations (color changes, precipitation formation, etc.) in relation to equilibria of systems studied

Procedure

Work in pairs on this experiment.

Each student must collect data and submit a separate report. The actual data analyses and the written reports must be done entirely independently of your lab partner or other students. Make sure that you avoid unauthorized collaboration and plagiarism. All suspected violations of the Code of Academic Conduct will be referred to Student Judicial Affairs.

Stock Chemicals Used

Chemical	Maximum Amount Used
6 M Hydrochloric Acid	< 15 mL
6 M Sodium Hydroxide	< 15 mL
6 M Acetic Acid	< 5 mL
6 M Ammonium Hydroxide	< 15 mL
1 M Ammonium Chloride	Drops
0.1 M Potassium Thiocyanate	< 5 mL
0.2 M Ferric Nitrate	< 5 mL
1 M Sodium Acetate	< 5 mL
0.5 M Oxalic Acid	< 5 mL
0.2 M Potassium Oxalate	< 5 mL
0.1 M Calcium Chloride	<5 mL
1% Cobalt Chloride in 95% Ethanol	< 10 mL
1% Phenolphthalein Indicator	Drops
1% Methyl Orange Indicator	Drops

Safety First

Remember to wear gloves and use caution whenever handling acids and bases.

Wear your goggles!

Before Starting the Experiment:

- Prepare data tables to record your observations. For part I, you may use the sample table given in the Data Analysis section, then use it as a guide to create your own data tables for the rest of the experiment.

Part I. Equilibria of Complex Ions

In this procedure, you will study the properties of a chemical system containing a complex ion. Many metal ions will bond with ions and molecules to form species called complex ions. An example of such a system is the combination of iron(III) ion with thiocyanate ion (SCN^-). When these two species are mixed they establish an equilibrium in water which can be described as:

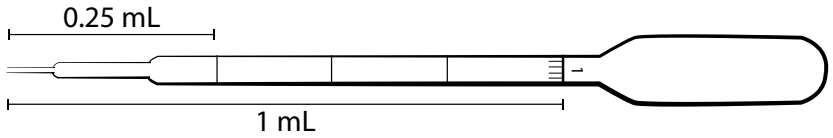
$$Fe^{3+}(aq) + SCN^-(aq) \rightleftharpoons FeSCN^{2+}(aq)$$

(pale yellow) **(deep red)**

Thus, the system will change color depending on the quantity of the complex ion present. In this part of the experiment, you will observe the change in the equilibrium position by adding various chemicals.

Estimating Volume with a Disposable Transfer Pipet

Because this experiment is mostly qualitative, you may use the disposable polypropylene transfer pipet to estimate the volume of reagents used.



To draw 1mL of solution into an empty disposable transfer pipet:

1. Squeeze the bulb to remove some air from the transfer pipet.
2. Submerge the tip of the transfer pipet in the solution.
3. Slowly release the pressure on the bulb and draw solution to the 1 mL mark.
4. Without releasing pressure on the bulb, steadily remove the pipet from the solution.
5. Release pressure on the bulb, allow the solution to enter the bulb of the pipet.
6. Place the pipet inside the receiving vessel, squeeze the bulb and release the solution.

Safety First

Keep chemical bottles in the spill tray and cap them when you are finished using them.

In case of spills on hands, change gloves immediately.

Recording Observations

Remember to record detailed observations on the changes you see! For more help with this, see the data analysis section of Observing Chemical Reactions.

1. Place 1 mL of 0.2 M $Fe(NO_3)_3$ and 1 mL of 0.1 M KSCN in two different test tubes. These will serve as your stock solutions and allow you to work from your bench without having to retrieve more.
2. Preparation of the $Fe(SCN)^{2+}$ solution:
   a. Add 12 mL of DI water to a 50 mL Erlenmeyer flask. To this flask, add 10 drops of 0.2 M $Fe(NO_3)_3$ and 5 drops of 0.1 M KSCN from the 2 stock solution test tubes you prepared in step 1. Mix the solution in the Erlenmeyer well with a disposable pipet. Record the color of the solution.
   b. Make sure that you can see through the solution; if the color is too dark you will have trouble observing any color changes as you proceed. You may continue to dilute the solution if it looks too dark. The volume given here is a general guideline.
   c. Place four 3 mL portions of this solution into four separate test tubes.
3. Add different reagents to the test tubes containing the $Fe(SCN)^{2+}$ solution (**NOT** the test tubes with the stock solutions from step 1) and record any color changes observed:

a. To the first test tube of $Fe(SCN)^{2+}$ add 0.5 mL of 0.2 M $Fe(NO_3)_3$ from the stock solution test tube.

b. To the second test tube of $Fe(SCN)^{2+}$ add 0.5 mL of 0.1 M KSCN from the stock solution test tube.

c. To the third test tube of $Fe(SCN)^{2+}$ slowly add 6 M NaOH solution drop-wise. You will notice two changes. You should address this in Question A below.

d. Use the last test tube of $Fe(SCN)^{2+}$ as a reference for comparison.

Question A: Compare the color of the solution in each of the four test tubes. Explain the color changes in terms of the equilibria described above and what you believe happened in Step 3.

Clean-Up

- Pour the content from the test tubes into the waste container in the fume hood labeled "Cation Metal Waste."

Part II. Equilibria of Acid/Base Indicators

Safety First

Do not rest your hands or arms on the edges of fume hood.

Always keep containers at least 6 inches away from the edge and inside the fume hood.

In this procedure, you will study the properties of two acid-base indicators, phenolphthalein and methyl orange. Many indicators are weak acids that establish equilibrium in water:

$$HIn(aq) + H_2O(l) \rightleftharpoons H_3O^+(aq) + In^-(aq)$$

(color 1) **(color 2)**

Thus, indicators can be thought of as dyes which change color depending on whether they are in a protonated (HIn) or unprotonated (In^-) form. In this part of the experiment you will observe the change in the equilibrium position of the indicators by adding acids and bases to solutions that contain these indicators.

Phenolphthalein indicator

1. Make sure you have approximately 5 mL of 6 M HCl in a test tube at the beginning of the experiment.

2. Place 3 mL of deionized water into each of three test tubes. Add two drops of phenolphthalein indicator to each of the test tubes. Observe the color of the solutions.

   a. To the first test tube, add two drops of 6 M HCl. Observe any color change.

   b. To the second test tube, add 4 drops of 6 M NaOH. Observe any color change.

   c. Use the third test tube as a reference.

3. Keep your remaining 6 M HCl for use in other parts of the experiment.

Methyl orange indicator

1. Place 3 mL of deionized water into each of three test tubes. Add two drops of methyl orange indicator to each of the test tubes. Observe the color of the solutions.

   a. To the first test tube, add two drops of 6 M HCl. Observe any color change.

   b. To the second test tube, add 4 drops of 6 M NaOH. Observe any color change.

   c. Use the third test tube as a reference.

Question B: What are the colors of the protonated and unprotonated forms of phenolphthalein?

Question C: What are the colors of the protonated and unprotonated forms of methyl orange?

Question D: Write the equilibrium expression for each indicator as shown above for HIn. Be sure to indicate the color of each form. Use Hph for the protonated form of phenolphthalein and Hmo for the protonated form of methyl orange.

Clean-Up

- Save the HCl solution until the end of the experiment.
- Using a small stream of no more than 5 mL of DI water total, rinse the contents of the other test tubes into an 800 mL beaker. Save this solution until Part IV.

Part III. Equilibria of Weak Acids and Bases

In this procedure, you will study the equilibrium properties of weak acids and bases. As described in the chapter on acids and bases, weak acids and bases establish equilibrium with water. In this part you will study this concept by using an acetic acid/acetate ion equilibrium system and the ammonia/ammonium ion equilibrium system. The pertinent equilibria for each system are:

$$HC_2H_3O_2(aq) + H_2O(l) \rightleftharpoons H_3O^+(aq) + C_2H_3O_2^-(aq)$$

$$NH_3(aq) + H_2O(l) \rightleftharpoons NH_4^+(aq) + OH^-(aq)$$

In this part of the experiment, you will observe the change in the equilibrium position through the use of the indicators used in Part I.

Procedure for the acetic acid/acetate ion equilibrium

1. Place 3 mL of 0.1 M acetic acid into each of three test tubes. You will make this solution from 6 M stock solution. Add two drops of methyl orange to each test tube. Observe the color of the solutions.

2. To one of these test tubes add 1.0 M $NaC_2H_3O_2$ a few drops at a time and observe any color changes. Remember to mix the solution well after each addition. To another test tube, add 6 M acetic acid a few drops at a time and observe any color changes. Again, mix well after each addition. Repeat this step with another sample to confirm your results.

Question E: Explain your observations using both the equilibria presented above and the one involving the indicator. What color change did you observe? How is the acetic acid/acetate ion equilibrium affected by adding acetate ion? How does this change affect the concentration of H_3O^+? How does the change in the concentration of H_3O^+ affect the H_{mo}/H_{mo-} equilibrium?

Procedure for the ammonia/ammonium ion equilibrium

3. Place 3 mL of 0.1 M ammonium hydroxide into the other three test tubes. You will make this from 6 M stock solution. Add two drops of phenolphthalein indicator to each tube. Observe the color of the solutions.

4. To one of these test tubes add 1 M NH_4Cl a few drops at a time and observe any color changes. Remember to mix the solution well after each addition. Add more 6 M ammonium hydroxide to the test tube a few drops at a time. Mix well after each addition and note any color changes.

Question F: Explain your observations using both the equilibria presented above and the one involving the indicator.

5. To another test tube containing ammonium hydroxide, add 6 M HCl a few drops at a time and observe any color changes. Remember to mix the solution well after each addition.

Question G: Explain your observations using both the equilibria presented above and the one involving the indicator.

Clean-Up

- Save the HCl solution until the end of the experiment.
- Using a small stream of no more than 5 mL of DI water total, rinse the contents of the other test tubes into the 800 mL beaker.
- Dissolve approximately 1 gram of sodium bicarbonate in the solution in the beaker. Dispose of the solution in the sink with copious amounts of water.

Part IV. Temperature Effects on Equilibria

In this procedure, you will again study the properties of a chemical system containing a complex ion. The system in this study of the temperature effects on equilibria can be described as:

$$Co(EtOH)_2Cl_2(aq) + 6H_2O(l) \rightleftharpoons 2EtOH(l) + 2Cl^-(aq) + Co(H_2O)_6^{2+}(aq) + heat$$

(blue) **(red-pink)**

Safety First

Ethanol is a flammable liquid. Keep the test tube capped and keep the test tube away from sources of ignition.

EtOH is the abbreviation for ethanol—CH_3CH_2OH. You will note that "heat" has been shown to be a "product" of the reaction as it is read from left to right. In other words, as the reaction occurs from left to right, the system gives off energy in the form of heat. Thus, the system will change color depending on whether heat is added or removed from the system.

1. Fill a 400 mL beaker half way with ice. Add a small amount of water.
2. Acquire a capped test tube containing 1% cobalt chloride dissolved in 95% ethanol. Place the test tube inside the ice bath, be careful to leave half the solution above the ice. Observe the color change.
3. Remove the test tube from the ice water bath and allow it to warm back to room temperature. Observe the color change.
4. Return the test tube to your TA.

Question H: Explain the color changes in terms of the equilibria described above.

Part V. Equilibria of Precipitation Reactions

In this procedure, you will study the properties of two chemical systems involving the oxalate anion, $C_2O_4^{2-}(aq)$. The chemical sources of the oxalate ion are calcium oxalate, $CaC_2O_4(s)$ and the weak diprotic acid, oxalic acid, $H_2C_2O_4(aq)$. This system is particularly interesting because 3 simultaneous equilibria occur in water. The equilibria in water that are important can be described as:

$$Ca^{2+}(aq) + C_2O_4^{2-}(aq) \rightleftharpoons Ca_2C_2O_4(s)$$

$$H_2O(l) + H_2C_2O_4(aq) \rightleftharpoons HC_2O_4^{-}(aq) + H_3O^{+}(aq)$$

$$H_2O(l) + HC_2O_4^{-}(aq) \rightleftharpoons C_2O_4^{2-}(aq) + H_3O^{+}(aq)$$

Clearly, this is a more complex system than we have thus far encountered. However, we should be able to qualitatively understand the system. For example, if you wanted to precipitate the calcium with oxalate, would you want the solution to be basic or acidic based on the equilibria described above? Take a guess! Now let's see if you are right.

1. Mix 4 mL of 0.1 M $CaCl_2$ with 4 mL of deionized water in a small beaker. Split the resulting solution into three approximately equal portions in three separate test tubes.

2. Add 15 drops of 0.2 M $K_2C_2O_4$ solution to one of the test tubes containing calcium chloride. Observe the results.

3. Add 6 drops of 0.5 M $H_2C_2O_4$ solution to one of the other test tubes containing calcium chloride. Observe the results.

Question I: Even though you added the same stoichiometric amount of oxalate ion to these test tubes you have observed differing amounts of precipitate. Why?

4. Now add 10 drops of 6 M HCl to the solution of calcium chloride and oxalic acid. Observe the results.

Question J: Explain the results in terms the equilibria discussed above.

5. Now slowly add 20 drops of 6 M NH_4OH to this test tube until a change occurs.

Question K: Explain the results in terms the equilibria discussed above.

6. You may wonder if the precipitate in Step 5 is really an oxalate or a hydroxide precipitate. This can be checked by adding 20 drops of 6 M NH_4OH to the solution in the last remaining test tube containing calcium chloride. Observe the results.

Question L: Do you believe the precipitate is calcium oxalate or calcium hydroxide? Explain.

Clean-Up

- Using a small stream of no more than 5 mL of DI water total, rinse the contents of all test tubes into the 800 mL beaker.
- Dissolve approximately 1 gram of sodium bicarbonate in the solution in the beaker.
- Dispose of the solution in the sink with copious amount of water.

Data Analysis

Data Table Preparation & Recording Observations

Observations will be the primary data collected in this qualitative lab. An example of a table for Part I can be found below. You may use this table for Part I, then use it as a guide for making your own tables for the other parts of this experiment.

Part I. Observation Table for Part I-Equilibria of Complex Ions

Test tube #	Contents	Procedure	Observations
1	3 mL dilute $Fe(SCN)^{2+}$	add 0.5 mL of 0.2 M $Fe(NO_3)_3$	
2	3 mL dilute $Fe(SCN)^{2+}$	add 0.5 mL of 0.1 M KSCN	
3	3 mL dilute $Fe(SCN)^{2+}$	add 6 M NaOH dropwise	
4	3 mL dilute $Fe(SCN)^{2+}$	reference	

Record your detailed observation of what happens in each test tube. Note any color change, precipitation, change in temperature, or bubbling.

- For more information on recording observations, refer back to the Data Analysis section of Observing Chemical Reactions.
- Make note of any reference samples, and remember that depending on how you create your table, not every cell will be filled in depending on what the procedure entails.

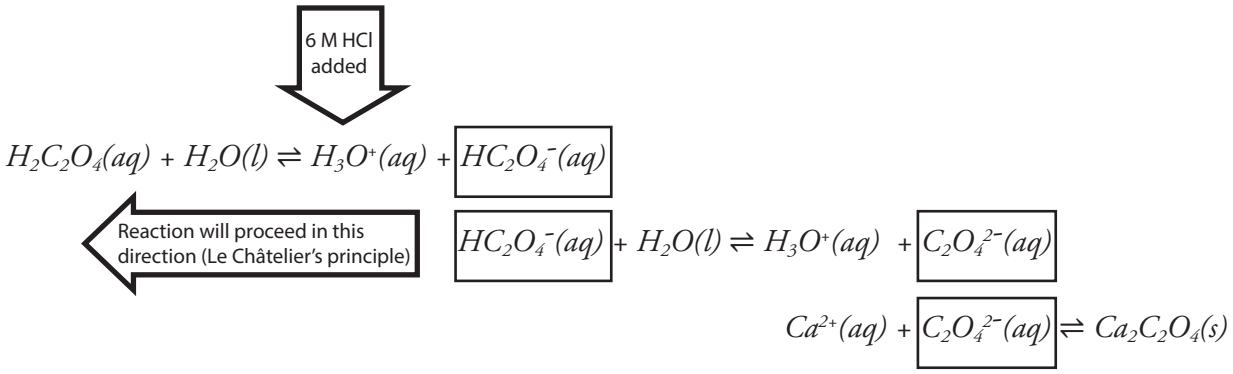
Le Châtelier's Principle

The equilibrium reactions observed in this lab can be explained using Le Châtelier's principle. If a chemical system is "stressed" via the introduction of excess reactants/products, change in pressure, or change in temperature, the system will adjust in order to re-establish equilibrium. To understand how stress affects a system, follow the procedure below:

1. Write out the chemical reaction in question.
    - Tip: Endothermic reactions require heat to proceed, so heat is listed as a reactant. Exothermic reactions generate heat, therefore heat is a product for these reactions.
2. Indicate which chemicals are being added to the system.
3. Draw an arrow indicating the overall direction that the reaction will proceed in order to relieve the stress of the added chemicals.
4. Consider how the shift will change the concentrations of both reactants and products, including heat, if applicable.

- Tip: Concentration is often indicated by writing brackets around a chemical formula (i.e. [HCl] means "the concentration of HCl").

Below is an example of this process applied to the equilibrium of oxalic acid after the addition of 6 M HCl:



From this analysis, we can see that when 6 M HCl is added, in order to re-establish equilibrium:

1. Equilibrium shifts to the left.
2. $[H_2C_2O_4]$ increases.
3. $[H_3O^+]$ decreases.
4. $[HC_2O_4^-]$ decreases.

The oxalate ion equilibrium in Part V is the most complex systems in this experiment. Changes in concentrations of one component (particularly something that appears as a product in one equation and as a reactant in another) will affect the equilibria of the whole system.

Strong Acid–Strong Base Titration

Introduction

Standardization

These next four experiments permit you to explore important aspects of acid-base chemistry. We start by exploring a straightforward reaction between a strong acid and strong base. The solutions you prepare in the strong acid-strong base experiment will be used as standardized solutions when you explore the titration of a weak-acid, a polyprotic acid, and a buffered system. **Titration** is a technique used to determine the concentration of a substance in solution. For example, food chemists often use titrations to determine the sugar, salt, or vitamin content in a sample. A **standard** solution is one whose concentration is accurately known, and can be used to determine the concentration of other solutions.

In this experiment, you will prepare a few standard solutions. If a solute can be obtained in a very pure, stable, weighable form, a **primary standard** solution of it can be prepared directly. To prepare a primary standard, an accurately determined amount of the solute must be dissolved in the desired solvent with an accurately known final volume. Care must be taken to ensure that the solution is homogeneous and that it is at ambient temperature when the final adjustment of its volume is made.

If the desired reagent cannot be obtained in primary standard form, one can only prepare a secondary or tertiary standard solution of it. A **secondary or tertiary standard** solution is prepared by dissolving an approximate amount of the solute in the desired solvent to the desired final volume, and standardizing the solution. A reagent solution may be standardized in a few ways:

1.) By titration against a measured mass of a suitable primary standard substance;

2.) By titration against another reliably known secondary standard solution;

3.) By direct analysis for the reagent in question by some suitable non-titrimetric method such as spectroscopic analysis.

In this experiment, you will prepare a primary standard consisting of an accurately weighed mass of potassium hydrogen phthalate (KHP). You will use this primary standard to determine the concentration of a sodium hydroxide (NaOH) solution, which will then be used as a secondary standard to determine the concentration of a hydrochloric acid (HCl) solution. The HCl solution will become your tertiary standard. Solid sodium hydroxide and hydrochloric acid are hygroscopic (i.e. they attract and hold water molecules from the surrounding environment), which makes it difficult to accurately weigh and determine molar amounts in a sample. For this reason they must be standardized against a primary or secondary standard.

Titrations

In this experiment you will determine the concentration of sodium hydroxide and hydrochloric acid solutions using a titration. The titration of HCl with NaOH takes advantage of the neutralization

reaction between a strong acid and a strong base. According to the **Arrhenius Acid-base Theory**, when acids and bases dissociate, or "ionize", in water, an acid raises the concentration of hydrogen ion, H^+, and a base raises the concentration of hydroxide ion, OH^-. When reacted together, the acid and base will neutralize each other according to net ionic equation (1). Note that H^+ can also be written as H_3O^+, as a result of H^+ associating with H_2O.

$$H^+(aq) + OH^-(aq) \rightarrow H_2O(l) \qquad (1)$$

An acid or base is considered strong if it completely ionizes in water. Thinking back to experiment 3, Chemical Equilibrium, this means the chemical equilibrium of a strong acid or base lays completely to the right of its dissociation equation. In this lab you will be utilizing the strong base sodium hydroxide to neutralize the strong acid hydrochloric acid according to the neutralization reaction below.

$$HCl(aq) + NaOH(aq) \rightarrow NaCl(aq) + H_2O(l) \qquad (2)$$

The progression of the reaction will be observed using a pH meter and a titration curve will be created using the experimental data. You will start with a sample containing acid and slowly add your standardized base. A **titration curve** is simply a plot of the pH of an acid versus the volume of base added, or vice versa.

The titration curve gives a good description of how an acid-base reaction proceeds. The pH will start out low and acidic, then increase as it approaches the equivalence point. The **equivalence point** is where the concentration of acid equals that of the base. If you know the concentration of one of these components, you can determine the concentration of the other using the equivalence point. As more base is added, the pH of the solution will continue to rise and then level off.

The progression of the reaction will also be monitored using the chemical indicator phenolphthalein, which turns pink in the presence of a basic solution. This color change occurs at the **endpoint** of the titration. It is important to note that the endpoint **does not** always correspond to the equivalence point, as phenolphthalein changes color in a basic solution, where the concentration of base has exceeded the concentration of acid.

What powers your lead storage batteries?

Lead storage batteries work by using acid-base reactions to generate electricity. The battery has two electrodes, a Pb anode and a PbO_2 cathode, which are submerged in an electrolyte solution of sulfuric acid. When the battery is discharged, the Pb anode and PbO_2 cathode react with the sulfuric acid to form lead(II) sulfate. In this reaction, the sulfuric acid is acting as a proton donor, or an acid. When the lead metal is oxidized to Pb^{2+}, it is acting as a Lewis base, or electron pair donor. The electrons flow from the anode to the cathode, which flow through an external circuit to create an electric current.

PbO_2 Cathode: $PbO_2(s) + 3H^+(aq) + HSO_4^-(aq) + 2e^- \rightarrow PbSO_4(s) + 2H_2O(l)$

Pb Anode: $Pb(s) + HSO_4^-(aq) \rightarrow PbSO_4(s) + H^+(aq) + 2e^-$

When the battery is recharged, the lead sulfate is converted back into Pb and PbO_2. This reaction requires an input of electrical energy, which is provided by an external power source. Normally, the battery is continuously charged by the alternator. In the case of a dead battery, the battery is charged by connecting it to a battery of a running automobile.

The acid-base reactions that occur in a lead battery are reversible, which enables its repeated usage in applications like vehicles and backup systems. However, the battery will eventually wear out and need to be replaced. The lifespan of a lead battery can vary depending on how it is used and maintained.

Learning Goals

The following is a list of skills that you will use in this experiment.

Laboratory	• Calculating volume and mass of reagents used to prepare solutions • Weighing by difference • Using a buret • Using a pH meter
Conceptual	• Arrhenius-Acid-Base theory • Properties of strong acids and bases (i.e. complete dissociation)
Data Analysis	• Properties of a strong acid/base titration curve • Using a spreadsheet to make a titration curve • Determining molarity of a titrated analyte • Statistical analysis (Q-test, standard deviation, 90% confidence limit)

Procedure

Work in pairs on this experiment.

Each student must collect data and submit a separate report. The actual data analyses and the written reports must be done entirely independently of your lab partner or other students. Make sure that you avoid unauthorized collaboration and plagiarism. All suspected violations of the Code of Academic Conduct will be referred to Student Judicial Affairs.

Stock Chemicals Used

Chemical	Maximum Amount Used
Potassium acid phthalate (KHP)	2.5 g
6 M Hydrochloric Acid	According to calculation (< 30 mL)
6 M Sodium Hydroxide	According to calculation (< 30 mL)
1% Phenolphthalein Indicator	Drops
pH Meter Calibration Buffer, pH 4 (red)	< 5 mL
pH Meter Calibration Buffer, pH 7 (yellow)	< 5 mL
pH Meter Calibration Buffer, pH 10 (blue)	< 5 mL

Safety First

Remember to wear gloves and use caution whenever handling acids and bases.

Wear your goggles!

Part I. Preparing your Solutions

You will prepare about 500 mL of approximately 0.2 M sodium hydroxide solution and 500 mL of approximately 0.2 M hydrochloric acid from 6.0 M stock solutions. Perform this calculation in the lab note book as part of the pre-lab exercise.

Hint

When determining the amount of concentrated stock solution needed to create a diluted solution, use $M_1V_1=M_2V_2$.

1. Preparing the 0.2 M NaOH solution:
   a. Label a 1 L bottle as 0.2 M NaOH.
   b. Estimate approximately 400 mL of DI water using a beaker and transfer it to the 1 liter plastic bottle.
   c. Estimate the volume of 6.0 M NaOH needed using a graduated cylinder, then quantitatively transfer the sodium hydroxide to a clean 150 mL beaker using a water bottle filled with DI water.
   d. Add DI water until the volume reaches 100 mL. Transfer this solution to the 1 liter plastic bottle.
   e. Cap the bottle securely, and mix the contents thoroughly by inverting the bottle and swirling it repeatedly. The bottle should be shaken at least 50 times in total.

2. Preparing the 0.2 M HCl solution:

   a. Label another 1 L bottle as 0.2 M HCl.

   b. Measure approximately 400 mL of DI water using a beaker and transfer it to the 1 liter plastic bottle.

   c. Calculate the volume of 6.0 M HCl needed to prepare your 0.2 M HCl solution.

   d. Using a beaker, obtain 6.0 M HCl and bring it back to your lab bench. At your bench, use a graduated cylinder to measure out the calculated amount of HCl. Quantitatively transfer the HCl to a clean 150 mL beaker using a water bottle filled with DI water.

   e. Add DI water to the 150 mL beaker until the volume reaches 100 mL. Transfer this solution to the 1 liter plastic bottle.

   f. Cap the bottle securely, and mix the contents thoroughly by inverting the bottle and swirling it repeatedly. The bottle should be shaken at least 50 times in total.

Safety First

Always add acid to water, and never the reverse.

Clean-Up:

- Using less than 5 mL of DI water, rinse any excess 6.0 M HCl and 6.0 M NaOH into an 800 mL beaker.
- Save this solution for the clean-up procedure at the end of lab.

Part II. Standardizing the base against Potassium Acid Phthalate

In this step of the experiment, you will standardize your sodium hydroxide solution against the primary standard, potassium acid phthalate, KHP. You will also use a technique called weighing by difference. This is a very important technique to use because it eliminates systematic errors from the balance. Be sure to use the same balance so that systematic errors in the balance will continue to be eliminated when you take the difference readings between masses.

Weighing by difference

- This technique eliminates systematic errors from the balance during weighing.
- First, measure the mass of the container with the material from which you are going to draw your sample. Then, remove some of the material and place it in a separate container. Re-measure the mass of the original container and the remaining material.
- Calculate the mass removed, and repeat the process until you have removed the mass desired.

Definitions

Titrant: a solution of known concentration used to determine the concentration of an analyte.

Analyte: a solution whose concentration is being measured.

1. Prepare the KHP samples:

   a. Accurately weigh a 0.5–0.6 g sample of dry KHP onto a weighing boat. Using the appearance of this sample as a guide, accurately weigh by difference 3 more samples onto weighing boats.

   b. Quantitatively transfer the first sample from the weighing boat into a 250 mL beaker with the help of a small stream of DI water from your wash bottle, and then add water to a total of about 30 mL and swirl it gently until the KHP dissolves. Transfer the other 3 samples of KHP into small Erlenmeyer flasks using the same procedure.

2. Prepare the buret:

   a. Read and understand how to fill and use a buret by reading the Common Laboratory Procedures section in the Appendix of this manual. Failure to fill the buret properly can result in spills and injuries.

Tips on using a buret

- Do not waste time trying to hit 0.00 with the meniscus. Fill the buret to slightly below the zero mark and read and record the actual starting point to the nearest 0.02 mL.
- Be careful when filling the buret. Only one person should be filling the buret. Be sure the stopcock is closed before filling.
- Use a 100 or 150mL beaker to fill the buret. **Never** use flasks, 1L plastic bottles, or large beakers to fill the buret.
- Be sure always to wipe off the tip of a buret before you begin a titration. Use a laboratory tissue and make one quick stroke downward beginning at the stopcock and ending in the air beyond the buret tip.

   b. Condition a 25 mL buret with your standardized NaOH solution. Remember to check that the stopcock is closed before filling a buret. While holding the buret at a safe level over the sink, use a beaker to pour in your sodium hydroxide solution.

   c. After conditioning, fill the buret to above the zero mark with a beaker, dispel any air bubbles from the stopcock, and place the buret in the buret clamp. Record the initial buret reading to two decimal places, eg. 1.24 mL.

3. Set up your titration apparatus according to Figure 10 Titration Setup in the Appendix.

    a. Place the stir plate underneath the buret. Adjust the clamp height so that there's enough room to place the KHP beaker directly under the buret.

    b. **Add three drops of phenolphthalein indicator and a stir bar to the first KHP solution.**

    c. Place the beaker onto the stir plate underneath the buret and turn on the stirrer and slowly increase the stirring speed. Don't use the heat control knob on the hot plate. Lower the buret tip well into the 250 mL beaker.

4. Perform a cursory titration using your KHP sample in a 250 mL beaker.

    a. Record the initial buret reading.

    b. Quickly add NaOH to the KHP beaker until the solution turns pink to determine the approximate endpoint of the titration. If the masses of KHP are almost the same across samples, then the endpoint of each sample will occur at approximately the same volume.

    c. After you have reached your endpoint for your cursory titration, record the buret reading at the endpoint and calculate the volume of NaOH needed to reach the endpoint.

    d. Retrieve, clean, and dry your magnetic stir bar for your next titration using the magnetic rod at the center sink. Pour the solution in the titration flask into an 800 mL beaker.

5. Perform a series of duplicate, precise titrations:

    a. From your cursory titration, you know the approximate position of the endpoint. Now you will perform a more precise titration using the following guidelines.

    b. Initially add the titrant fairly rapidly, pausing every few milliliters to allow the solution to mix thoroughly. Pay attention to the region where the two solutions mix and as the indicator color begins to tail out into the solution as you stir, reduce the next amount of titrant added, keeping in mind the target volume. Stop adding titrant about 1 mL short of this volume.

    c. Gently wash down the walls of the flask with water from your wash bottle, and then resume adding base from the buret but now dropwise. As you approach the endpoint, the pink color will increasingly linger. You should frequently wash down the interior sides of the flask to recover any reagent drops that may be clinging to the sides. Stop adding base when the entire flask has a ***faint*** pink color that persists.

Lab Skill Tips

Read the buret at or slightly below eye level. Do not hold your hands or lab manual behind the buret.

You can hold a sheet of colored paper behind the buret to read the meniscus clearly.

d. You may wish to record the buret volume of several successive drops as you approach the endpoint in case you discover that you have overshot the endpoint. Record the final buret reading to the nearest 0.02 mL.

e. Refill the buret and similarly titrate the remaining two KHP samples.

f. Clean up by pouring the solution in the titration flasks into your 800 mL beaker.

6. Calculating the concentration of your NaOH solution:

   a. Since NaOH and KHP react in a 1:1 molar ratio, the equivalence point occurs when the moles of NaOH added to the flask equals the moles of KHP present.

   b. The number of moles of KHP present in the flask can be determined using the mass of KHP added to the flask.

   c. Calculate the molarity of your prepared NaOH solution using the moles and volume of NaOH added to the flask at the equivalence point.

   d. Determine the molarity of NaOH for all three titrations.

7. Following directions found in the appendix, perform a Q-test on the calculated molarities from all three titrations.

   a. If a value fails the Q-test, perform another titration and discard the outlier.

   b. You should discard the data from the first cursory titration if this titration was performed quickly.

Part III. Strong Acid-Strong Base Titration Curve

This part of the experiment requires the use of a pH meter to measure the pH of various solutions. The pH meter and the accompanying electrode are both very expensive and fragile. Treat both pieces of equipment with great care. Read and understand the pH meter operation instructions in the Common Laboratory Procedures section of Appendix of this manual. Follow the directions provided very carefully.

Care of pH meter and pH electrodes

- Keep the tip of the electrode submerged in solution at all times.
- Never leave the electrode in deionized water.
- When rinsing the electrode use a light stream of deionized water.
- Be careful of the electrode when adding strong base or when stirring the solution. Do not stir using the electrode.
- After completing the experiment, **store the electrode in the storage solution provided.** Additional storage solution is available in the laboratory if needed.

The information you generate in this part of the experiment has two goals: 1) to standardize the approximately 0.2 M HCl solution, and 2) to demonstrate the classic titration curve of acid-base chemistry. You will be doing the titration procedure at least twice. The first titration will familiarize you with the critical pH and volumes for your specific solutions' concentrations, after which you may adjust your technique to more accurately locate the endpoint for the specific solutions you are using.

1. Calibrate the pH meter.

   a. The pH meter will need to be calibrated before starting the experiment. There is no need to recalibrate later during the experiment.

   b. Dispense the pH 4.00, pH 7.00, and pH 10.00 buffers into the mini buffer bottles, taking care not to overfill.

      Standardize the pH meter using the three buffer solutions following the procedure outlined in the Appendix of this manual, pH Meter Operating Instructions.

      Always keep the pH electrode in the electrode storage solution when not in use.

2. Prepare the titration vessel.

   a. Use your volumetric pipets to quantitatively transfer 15.00 mL of the approximately 0.2 M HCl solution you prepared in Part I into a 150 mL beaker. Without measuring precisely, add about 15 mL of deionized water to this beaker. Place a magnetic stir bar into the beaker gently so no reagent splashes.

   b. Place a magnetic stir plate under a buret clamp that is adjacent to the pH meter you have just standardized near your work area and place the beaker on the stir plate.

   c. Use the previously conditioned buret from Part II filled with the approximately 0.2 M NaOH solution that you standardized in Part II and clamp it in place above the beaker.

d. Clamp the pH electrode in place below the level of the liquid in the beaker and away from the stir bar. Adjust the position of the buret tip so that it is inside the beaker, away from the side but with the stopcock at a convenient location for you to manipulate. Position the beaker so that the stir bar is somewhat displaced away from the center of the beaker to allow room for the pH electrode but make sure that the stir bar is above the center of the stir plate.

3. You will now standardize the HCl solution by titrating it with the previously standardized NaOH solution, and following the course of the acid neutralization reaction by monitoring the pH with the pH meter.

Tip

When recording data on a spreadsheet, leave an empty column between the buret reading and the corresponding pH. This column can be used to calculate the total volume of NaOH added (the difference between present buret reading and initial buret reading).

4. Begin with a quick titration.

   To do so, you will add the titrant (0.2 M NaOH) in small increments, and recording the volume reading in the buret and the pH reading from the pH meter in your note book.

   Follow the instructions closely and add the titrant in the correct increments.

   a. When the assembly is complete, turn on the stir motor (left knob) slowly so that the stir bar is rotating at a smooth, moderate speed and clears the pH electrode. Do **NOT** turn on the heat.

   b. Perform a quick titration by adding the titrant in 1 mL increments until you reach pH 2.5; then 0.10 mL (2 drops) increments until you reach pH 10.7. After that, add 1 mL increments until pH 11.5. Stop the titration when you have reached pH > 11.5.

   c. When the titration is complete, pour the solution from the titration vessel into the 800 mL beaker.

Titration Tips

- **DO NOT** use your wash bottle to rinse down the sides of the beaker, as any added volume will change the pH readings and invalidate the titration curve data being collected.
- Record your buret readings after the addition of each increment.
- Allow time for the reaction vessel to become equilibrated and for the pH reading to become stabilized and then record the pH value in your notebook alongside the buret reading.

   d. To find the endpoint, first convert your buret readings to volumes of NaOH added. Determine between which 2 volumes the largest change in pH occurs. The **endpoint** is between the volume readings where the largest pH change occurred.

5. Set up your second titration by repeating step 2.

6. Your second titration should be more carried out more precisely.

   Refine your procedure based on your first titration by adding 1 drop of NaOH at a time from well below the endpoint to well above the endpoint. Record your buret readings after the addition of each increment.

   Allow time for the reaction vessel to become equilibrated and for the pH reading to become stabilized and then record the pH value in your notebook alongside the buret reading.

   a. Turn on the stir motor (left knob) slowly so that the stir bar is rotating at a smooth, moderate speed. If necessary, adjust the position of the pH electrode so that it does not touch the stir bar. Do **NOT** turn on the heat.

   b. Begin by adding the titrant in 1 mL increments until you are approximately 2 mL away from the endpoint. Record the volume and pH reading at each increment.

   c. Now, add the titrant in drop-wise increments. Stop the titration when you have reached pH > 11.5.

   d. When the titration is complete, pour the solution from the titration vessel into the 800 mL beaker.

7. Repeat the titration procedure as time allows so that you have as many trials as possible to improve the statistics of your standardization of HCl.

Clean-Up

- Tightly cap and store the bottles containing the standardized NaOH and HCl solutions for use in later experiments.
- Drain the remaining NaOH from the buret into the 800 mL beaker. Add any left-over KHP, and any excess 6 M HCl into the 800 mL beaker.
- Slowly and carefully, add 1 gram of sodium bicarbonate to the solution in the 800 mL beaker.
- Pour the solution into the sink with copious amount of water.

► **SAVE your standardized 0.2 M HCl and 0.2 M NaOH. You will use these solutions for the next 3 experiments.**

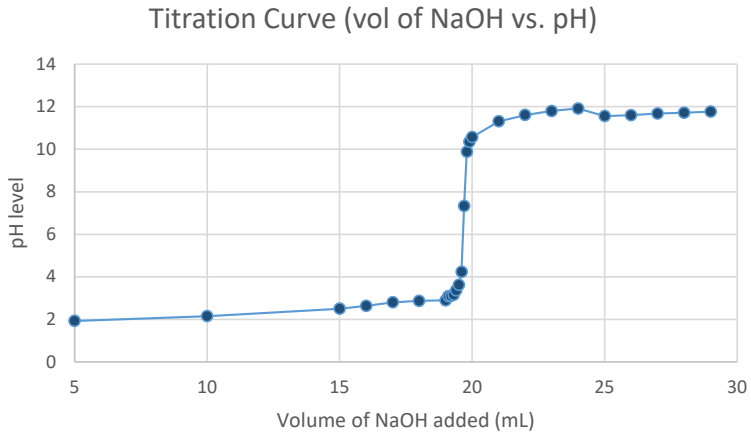
Data Analysis

Calculating concentration of NaOH solution

1. Using the mass of KHP you weighed out in part 2, determine how many moles of KHP were present in the sample for your first good titration.
    - Tip: KHP has the chemical formula $KHC_8H_4O_4$, with a formula mass of 204.23 g/mol.
2. Write down the chemical equation for the neutralization reaction of NaOH with KHP. Determine the stoichiometric ratio between these two reagents. In other words, how many moles of NaOH react with each mole of KHP?
3. Using the information you found in steps 1 and 2, determine how many moles of NaOH were dispensed in your first titration at the equivalence point.
    - Tip: Keep in mind the equivalence point occurs when the KHP is fully neutralized by NaOH. This means all moles of KHP will have reacted with NaOH.
4. Using the moles of NaOH calculated in step 3 and the volume of NaOH added at the equivalence point, determine the molarity of your NaOH solution. Remember to pay attention to units!
    - Tip: When calculating the volume of NaOH added, take the difference between the equivalence point buret reading and initial buret reading.
5. Repeat steps 1-4 to calculate the molarity of your NaOH for each titration.
6. Calculate the average molarity of your NaOH solution. This solution has now been "standardized", and can be used as a titrant to determine the concentration of other analytes.
7. Calculate the standard deviation of the average molarity of your NaOH solution.
8. Calculate the 90% confidence limit for your average molarity.

Generating titration curves

9. Use a spreadsheet program such as Excel to enter the buret readings and corresponding pH for each titration. Use the buret readings to calculate the total volume of NaOH added at each point in the titration.
    - Tip: The total volume of NaOH added at any point in the titration can be calculated by taking the difference between that buret reading and the initial buret reading.
10. Generate a scatter plot of the total volume of NaOH added vs. pH. Make sure to include a plot title and labeled axes with units.
11. Repeat steps 9 and 10 for each titration of NaOH with HCl.



Generating 1st and 2nd derivative curves

As you'll hopefully see from your titration curves, the pH of a solution is much more sensitive near the equivalence point. This means that even a small volume of titrant can have a great effect on the pH. Sometimes it is difficult to determine the exact volume of titrant at which the equilibrium point occurs just from using the titration curve. To help determine this volume, we will construct approximations of the 1st and 2nd derivative plots of the original titration curves. A derivative is a mathematical concept used to describe rate of change at a certain point. By finding the 1st derivative, we can see where the greatest change in pH occurs over the smallest amount of volume (i.e. where the slope of our titration curve is the steepest). By finding the 2nd derivative, we can see where there is an inflection point in the curve of the 1st derivative (the 2nd derivative will change sign). This point will be the equivalence point. It is important to note that this method of calculating derivatives, called "forward divided difference", does tend to amplify experimental error (commonly called noise), but your data should be good enough that these plots of the forward divided differences can help you to identify the equivalence point.

1. For your first good titration, label the column containing total volume of NaOH added as "V" and the column containing the pH as "pH"

2. Leave four empty columns to the right of these two columns for calculating values for the derivatives. Label these columns as "Vm" (midpoint volume), "D1" (1st forward divided difference), "Vd" (derivative midpoint volume), and "D2" (2nd forward divided difference). Vm and D1 will be used to plot the 1st derivative, and Vd and D2 will be used to plot the 2nd derivative.

3. We will start by calculating values for Vm. Vm is the midpoint between volumes of NaOH added. For example, if the first addition of NaOH was 1.00 mL, and then you added another 1.00 mL (for a total of 2.00 mL), the midpoint volume would be 1.50 mL. This can be mathematically expressed using the following equation, where V_i is one of the volume data points and V_{i+1} is the next data point in the sequence.

$$Vm_i = \frac{(V_i + V_{i+1})}{2}$$

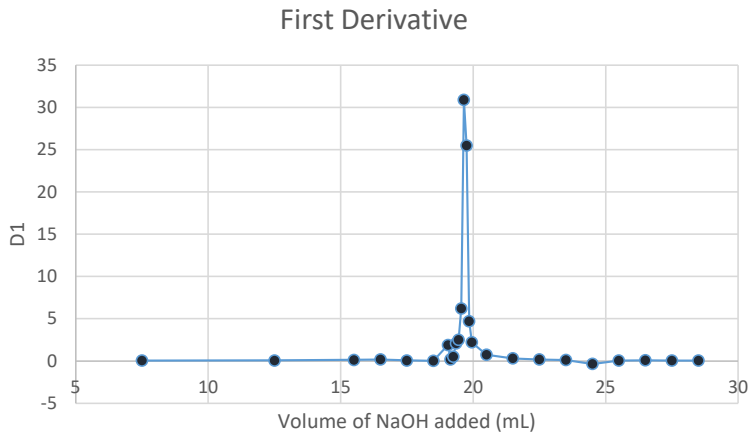
In the column labeled "Vm", enter the formula that will calculate the volume midway between V_i and V_{i+1}, but instead of "V_i", select the spreadsheet cell containing the first volume of NaOH, and instead of "V_{i+1}", select the cell containing the second volume of NaOH added. Apply this equation to the rest of the cells in the column by clicking in the bottom right-hand corner of the cell and dragging downwards.

4. Next we will calculate values for D1. D1 is the rate of change in pH as NaOH is added. This can be expressed using the following equation, where V_i and V_{i+1} are the same as in step 3, and pH_i and pH_{i+1} are the pH readings corresponding to those volumes.

$$D1_i = \frac{(pH_{i+1} - pH_i)}{(V_{i+1} - V_i)}$$

In the column labeled "D1", enter the formula for D1 using the cells containing data from the first and second volumes of NaOH added and the corresponding pHs. Apply this equation to the rest of the cells in the column. Notice that you will not be able to apply this calculation to the last row of the data since there are no data values beyond the last row to use for pH_{N+1} or V_{N+1}.

5. Generate a 1st derivative graph by plotting your calculated midpoint volumes (Vm) on the x-axis of a scatter plot, and the 1st forward divided differences (D1) on the y-axis. The equivalence point of the titration is the maximum point on this curve.



Now we will calculate the derivative midpoint volume, Vd. Vd is calculated the same way as Vm, but instead of finding the midpoint volume between original volumes of NaOH added, you will find the midpoint between Vm values. This can be mathematically expressed using the following equation, where Vm_i is one of the Vm data points and Vm_{i+1} is the next data point in the sequence.

$$Vd_i = \frac{(Vm_i + Vm_{i+1})}{2}$$

In the column labeled "Vd", enter this formula to calculate the volume midway between Vm_i and Vm_{i+1}. Just like with calculating Vm, select the appropriate spreadsheet cell containing the values for Vm_i and Vm_{i+1}. Apply this equation to the rest of the cells in the column by clicking

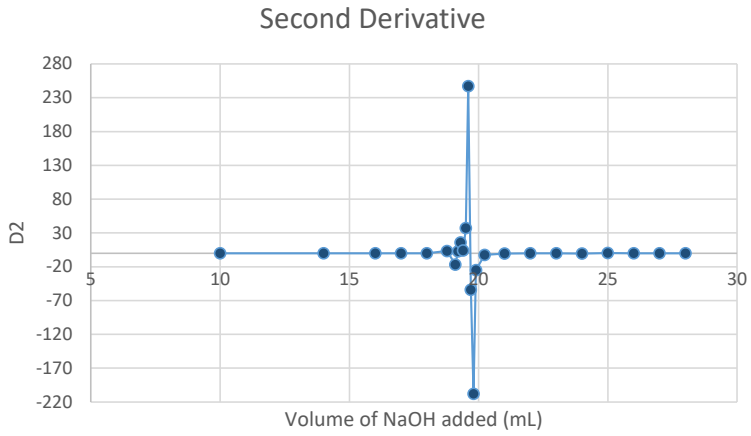
in the bottom right-hand corner of the cell and dragging downwards. Notice that you will not be able to apply this calculation to the last row of data.

6. Next we will calculate values for the 2nd forward divided difference, D2. D2 is calculated the same way as D1, but instead of finding the rate of change in pH, you will find the rate of change in D1. This can be expressed using the following equation, where Vm_i and Vm_{i+1} are the same as in step 3, and $D1_i$ and $D1_{i+1}$ are the 1st forward divided differences corresponding to those midpoint volumes.

$$D2_i = \frac{(D1_{i+1} - D1_i)}{(Vm_{i+1} - Vm_i)}$$

   In the column labeled "D2", enter this formula to calculate the 2nd forward divided difference. Just like with calculating D1, use the appropriate spreadsheet cells containing values for $D1_i$, $D1_{i+1}$, Vm_i and Vm_{i+1}. Apply this equation to the rest of the cells in the column. Notice that you will not be able to apply this calculation to the last row of data.

7. Generate a 2nd derivative graph by plotting your calculated derivative midpoint volumes (Vd) on the x-axis of a scatter plot, and the 2nd forward divided differences (D2) on the y-axis. The equivalence point of the titration is the value at which the plot passes through the x-axis..



8. Repeat steps 1-7 for the rest of your titrations.

   Check with your teaching assistant to see how they want the graphs turned in (i.e. by email or printed copies). Make sure your name is on each graph, and that you have included a title and labeled axes with units.

Calculating concentration of HCl solution using titration curves

In part II of this lab, KHP was used as a primary standard to determine the concentration of NaOH solution. This NaOH solution can now be used as a secondary standard to determine the concentration of your HCl:

1. Using the titration curves and derivative plots you previously developed, what is the equivalence point volume of NaOH for each of your titration curves? You should be able to determine this value within 0.02 mL (eg. 18.46 mL).
2. Using the **average molarity** of your NaOH solution, and the equivalence point volumes of NaOH determined from the derivative plots, calculate how many moles of NaOH were dispensed in each titration at the equivalence point.
3. Write down the chemical equation for the neutralization reaction of NaOH with HCl. Determine the stoichiometric ratio between these two reagents. In other words, how many moles of NaOH react with each mole of HCl?
4. Use the moles of NaOH dispensed and the stoichiometric ratio between NaOH and HCl to determine the number of moles of HCl present in each sample.
5. Using the number of moles of HCl and the volume of HCl in each sample, determine the molarity of HCl for each titration. Remember to pay attention to units!
6. Calculate the average molarity of your HCl solution. This solution has now been "standardized", and can be used as a titrant to determine the concentration of other analytes.
    - Keep the average values of your standardized NaOH and HCl solutions in a prominent place in your notebook and perhaps write the value on the bottle labels. You will need these values in subsequent experiments.
7. Calculate the standard deviation of the average molarity of your HCl solution.
8. Calculate the 90% confidence limit for your average molarity.

Sample Data and Plots

TITRATION EXAMPLE: Titration of 15 mL HCl with 0.20 M NaOH

NaOH (Vol added, mL)	pH	Vm	D1	Vd	D2
5.00	1.93	7.50	0.04	10.00	0.01
10.00	2.15	12.50	0.07	14.00	0.02
15.00	2.50	15.50	0.13	16.00	0.04
16.00	2.63	16.50	0.17	17.00	-0.10
17.00	2.80	17.50	0.07	18.00	-0.04
18.00	2.87	18.50	0.03	18.78	3.40
19.00	2.90	19.05	1.90	19.10	-17.00
19.10	3.09	19.15	0.20	19.20	3.00
19.20	3.11	19.25	0.50	19.30	16.00
19.30	3.16	19.35	2.10	19.40	4.00
19.40	3.37	19.45	2.50	19.50	37.00
19.50	3.62	19.55	6.20	19.60	247.00
19.60	4.24	19.65	30.90	19.70	-54.00
19.70	7.33	19.75	25.50	19.80	-208.00
19.80	9.88	19.85	4.70	19.90	-25.00
19.90	10.35	19.95	2.20	20.23	-2.65
20.00	10.57	20.50	0.74	21.00	-0.44
21.00	11.31	21.50	0.30	22.00	-0.11
22.00	11.61	22.50	0.19	23.00	-0.08
23.00	11.80	23.50	0.11	24.00	-0.46
24.00	11.91	24.50	-0.35	25.00	0.39
25.00	11.56	25.50	0.04	26.00	0.04
26.00	11.60	26.50	0.08	27.00	-0.04
27.00	11.68	27.50	0.04	28.00	0.01
28.00	11.72	28.50	0.05		
29.00	11.77				

Acid Dissociation Constants and the Titration of a Weak Acid

Introduction

One of the most important applications of equilibria is the chemistry of acids and bases. The **Brønsted-Lowry acid-base theory** defines an **acid** as a species that donates a proton and a **base** as a species that accepts a proton. In the case of an aqueous solution of a strong acid, such as HCl, the acid reacts completely with the water and dissociates into the hydronium ion, H_3O^+, and the chloride ion, Cl^- as shown by

$$HCl(aq) + H_2O(l) \rightarrow H_3O^+(aq) + Cl^-(aq) \qquad (1)$$

In this reaction, HCl is the **Brønsted-Lowry acid** and H_2O is the **Brønsted-Lowry base**. In an aqueous solution of HCl, the associated species, HCl, does not exist. The species present are H_3O^+, Cl^-, and H_2O. Since this reaction essentially goes to completion, a single-headed arrow pointing to the right is used in the chemical equation.

Unlike strong acids, aqueous solutions of weak acids **do not** completely dissociate into the hydronium ion and the corresponding anion but instead reach **equilibrium**. If we let "HA" symbolize a weak acid, then the equilibrium reaction of a weak acid with water is represented by

$$HA(aq) + H_2O(l) \rightleftharpoons H_3O^+(aq) + A^-(aq) \qquad (2)$$

Similarly, in this reaction, HA is the Brønsted-Lowry acid and H_2O is the Brønsted-Lowry base. In an aqueous solution of HA, the species present are the associated species, HA, the hydronium ion, H_3O^+, the anion, A^-, and H_2O. Note that double arrows pointing in opposite directions are used in the chemical equation since this reaction does not go to completion but instead reaches equilibrium. We explored multiple examples of weak acid equilibrium in the experiment Chemical Equilibrium.

HA and A^- are also referred to as a **conjugate acid-base pair** where HA is the acid and A^- is its conjugate base, formed when HA donates its proton. The species A^- is also considered to be a Brønsted-Lowry base since it can accept a proton. The species that make up a conjugate acid-base pair only differ in structure by the presence of a single proton, H^+. Likewise, H_2O and H_3O^+ also constitute a conjugate acid-base pair where H_3O^+ is the conjugate acid of H_2O.

Since equation (2) is an equilibrium reaction, we can write an equilibrium constant expression as shown below

$$K_a = \frac{[H_3O^+][A^-]}{[HA]} \qquad (3)$$

The equilibrium constant, K_a, is called the **acid dissociation constant**. Recall that water is not included in the equilibrium constant expression since it appears in the reaction as the pure

liquid. The magnitude of the dissociation constant provides information regarding the degree of dissociation of the acid in water. For example, the K_a values for HF and HCN are 7.2×10^{-4} and 4.0×10^{-10}, respectively. The larger K_a value of HF indicates that the equilibrium reaction between HF and H_2O (4) lies further to the right than the equilibrium reaction between HCN and H_2O (5).

$$HF(aq) + H_2O(l) \rightleftharpoons H_3O^+(aq) + F^-(aq) \qquad (4)$$

$$HCN(aq) + H_2O(l) \rightleftharpoons H_3O^+(aq) + CN^-(aq) \qquad (5)$$

In other words, HF dissociates into the hydronium ion, H_3O^+, and its conjugate base, F^-, to a greater extent than does HCN. If we had a bottle of 0.1 M HF and a bottle of 0.1 M HCN, then the hydronium ion concentration would be higher in the bottle of HF than in the bottle of HCN; therefore, the pH would be lower in the bottle of HF.

Due to the establishment of equilibrium between a weak acid and its conjugate base in an aqueous medium, the pH changes that take place when titrating a weak acid with a strong base are significantly different than the pH changes that take place when titrating a strong acid with a strong base. As a result, the titration curve of a weak acid has a slightly different shape than the titration curve of a strong acid. For example, when a strong acid is titrated with a strong base, the equivalence point is found to occur at pH = 7. However, when a weak acid is titrated with a strong base the equivalence point does not occur at neutral pH. You will also find other significant differences between the two titration curves due to equilibrium reactions.

Let us consider in more detail how pH will change when small amounts of strong base are added to an aqueous solution of a weak acid, HA. Before any strong base is added to the weak acid, the concentration of the hydronium ion can be assumed to originate only from the dissociation of the weak acid.

$$HA(aq) + H_2O(l) \rightleftharpoons H_3O^+(aq) + A^-(aq) \qquad (2)$$

The assumption here is that the amount of hydronium ion resulting from the dissociation of water

$$H_2O(l) + H_2O(l) \rightleftharpoons H_3O^+(aq) + OH^-(aq) \qquad (6)$$

is **very small** relative to the other sources of hydronium and can be neglected. This is a good assumption since the equilibrium constant for water, K_w, at 25°C is equal to 1.0×10^{-14}. Therefore, the pH corresponds to the $[H_3O^+]$ generated by the dissociation of HA.

Looking at equation (2), for every mole of H_3O^+ that forms, one mole of A^- is produced and one mole of HA dissociates. Therefore, at equilibrium, $[H_3O^+] = [A^-]$ and $[H_3O^+]$ represents the concentration of HA that is lost in the dissociation. Once the initial concentration of HA is known, then the equilibrium concentrations of H_3O^+, A^-, and HA can be calculated, as well as the K_a value of the weak acid from a measured pH value.

As base is added, the OH^- ion will react with the major species in solution, HA, to produce more conjugate base, A^-.

$$HA(aq) + OH^-(aq) \rightarrow H_2O(l)) + A^-(aq) \qquad (7)$$

This reaction can be assumed to go to completion followed by the re-establishment of the dissociation equilibrium:

$$HA(aq) + H_2O(l) \rightleftharpoons H_3O^+(aq) + A^-(aq) \qquad (2)$$

If the equivalence point has not been reached, then the number of moles of leftover HA will be equal to the original number of moles of HA minus the number of moles of OH^- added. The moles of HA lost in the dissociation reaction, shown by equation (2), will be negligible compared to the number of moles of leftover HA.

[HA] then is calculated by dividing the number of leftover moles of HA by the total volume of the mixture at this point in the titration. $[H_3O^+]$ is determined by the number of moles of H_3O^+ formed in the dissociation reaction and is simply measured by pH. (Again, assuming that any $[H_3O^+]$ formed from the dissociation of water is negligible.) $[A^-]$ is equal to the number of moles of A^- formed in the strong base reaction, shown by equation (7), divided by the total volume of the mixture. Like HA, the number of moles of A^- produced in the dissociation reaction is negligible.

Another point of interest on a weak acid titration curve, other than the equivalence point, is the **midpoint**. The midpoint occurs when ½ of the original acid, HA, has reacted with all the strong base, OH^-, that has been added. At the midpoint, the number of moles of the conjugate base, A^- is equal to the number of moles of weak acid, HA, remaining in the solution and thus, $[HA] = [A^-]$. Applying this to equation (3), we obtain $K_a = [H_3O^+]$ and taking the negative log of each side, the equality is expressed as $pH = pK_a$. Therefore, at the midpoint, the K_a of the weak acid can be easily calculated from the measured pH level.

At the equivalence point, just enough strong base has been added to completely react with all the weak acid. After the reaction, the only species present will be the conjugate base, A^-. Since A^- is a conjugate base, it will accept a proton from water to reform HA and OH^- in the equilibrium reaction shown by:

$$A^-(aq) + H_2O(l) \rightleftharpoons HA(aq) + OH^-(aq) \qquad (8)$$

The equilibrium constant expression is given by:

$$K_b = \frac{[HA^-][OH^-]}{[A^-]} \qquad (9)$$

The equilibrium constant, K_b, is called the **base dissociation constant**. Knowing the original amount of HA placed into the flask, measuring the pH, and making the assumption that the concentration of the OH^- is the same as the concentration of HA, you can determine the concentrations of all three of the species in this equilibrium constant expression.

The K_a of a weak acid and the K_b of the corresponding conjugate base are related to each other by the equilibrium constant, K_w.

$$K_w = K_a K_b \qquad (10)$$

By subtraction, you should be able to calculate the concentration of A^- in solution. Finally, using the relationship shown by equation (10), you will be able to re-calculate the value of K_a for the weak acid.

Beyond the equivalence point in the titration, the strong base, OH^-, will be in excess. Here, the excess base determines the pH of the solution. The amount of OH^- formed from the equilibrium reaction shown by equation (8) is negligible. You will then plot all the pH measurements made in this experiment against the quantity of strong base added to form a pH titration curve.

In this experiment, you will be titrating the weak acid, acetic acid, with the strong base, sodium hydroxide. After you find the volume of strong base needed to reach the equivalence point of the titration, you will use this information to calculate the concentration of the original weak acid solution. You will calculate the acid dissociation constant, K_a, of acetic acid using several measured pH readings along the titration. You will also compare the titration curves of a strong acid titration and weak acid titration.

Saponification: good for soap, bad for your skin

The process of making soap, called "saponification", involves a reaction between weak fatty acids from animals or plants, and lye, which is a solution of NaOH or KOH in water. The final soap product consists of a fatty acid salt, which cleans by attracting grease and dirt to the nonpolar end of the fatty acid chain, and water to the polar salt end. Saponification can also occur to the fatty acids in skin, turning them into soap, which is why it's important to be extra careful while handling strong bases!

Learning Goals

The following is a list of skills that you will use in this experiment.

Laboratory	• Calculating volume and mass of reagents used to prepare solutions • Performing a titration
Conceptual	• Properties of weak acids and bases (i.e. conjugate acid-base pairs, dissociation strength, equilibrium) • Brønsted-Lowry acid base theory
Data Analysis	• Properties of a weak acid/base titration curve • Calculating acid-base dissociation constants • ICE tables • Relationship between $[H_3O^+]$ and pH

Procedure

Work in pairs on this experiment.

Each student must collect data and submit a separate report. The actual data analyses and the written reports must be done entirely independently of your lab partner or other students. Make sure that you avoid unauthorized collaboration and plagiarism. All suspected violations of the Code of Academic Conduct will be referred to Student Judicial Affairs.

Preparation for Next Lab

Before starting the Weak Acid Titration experiment, each pair of students needs to dry a sample of solid sodium carbonate in preparation for next week's Polyprotic Acid experiment.

1. Fill one vial with approximately 1 g of anhydrous sodium carbonate.
2. Place the uncapped vial in your 100 mL beaker to keep the vial from spilling in the oven.
3. Dry the sample in the oven for 1.5 hours. Do not adjust the temperature on the oven. The temperature on the oven has been preset and will heat to the correct temperature when the door remains closed.
4. After removing your sample from the oven, let it cool until it is warm but safe to handle.
5. After the sample has cooled, carefully remove the vial from the beaker using a test tube clamp and place it in the center of the desiccator in your locker. If the lid of your desiccator can be removed easily, ask your TA for some vacuum grease to properly seal your desiccator.

Stock Chemicals Used

Chemical	Maximum Amount Used
6 M Acetic Acid	< 10 mL
0.05% Thymolphthalein Indicator	Drops
pH Meter Calibration Buffer, pH 4 (red)	< 5 mL
pH Meter Calibration Buffer, pH 7 (yellow)	< 5 mL
pH Meter Calibration Buffer, pH 10 (blue)	< 5 mL
Sodium Carbonate(s)	< 1 g

Safety First

Remember to always wear gloves when handling all acids and bases.

Wear your goggles!

Part I. Solution Preparation

1. Prepare 200 mL of approximately 0.05 M acetic acid solution from 6 M acetic acid.

   a. Calculate the volume of stock solution required in this dilution.

   b. Add the appropriate amount of 6 M acetic acid to approximately 150 mL of DI water in a 250 or 500 mL Erlenmeyer flask.

   c. Add DI water to the flask until the solution level reaches 200 mL.

   d. Mix well.

Safety First

Before inverting the NaOH bottle, make sure it is properly capped.

2. Find your 1 L bottle of your standardized NaOH from the previous experiment. Before opening the bottle of NaOH, carefully invert it several times to ensure that your solution is homogenized.

Part II. Weak Acid Strong Base Titration Curve

This experiment requires the use of a pH meter to measure the pH of various solutions. The pH meter and the accompanying electrode are both very expensive and fragile. Treat both pieces of equipment with great care.

Care of pH meter and pH electrodes

- Keep the tip of the electrode submerged in solution at all times.
- Never leave the electrode in deionized water.
- When rinsing the electrode use a light stream of deionized water.
- Be careful of the electrode when adding strong base or when stirring the solution.
- After completing the experiment, **store the electrode in the storage solution provided.** Additional storage solution is available in the laboratory if needed.

Titration Set Up

1. Calibrate the pH meter.

   a. The pH meter will need to be calibrated before starting the experiment. There is no need to recalibrate later during the experiment.

   b. Dispense the pH 4.00, pH 7.00, and pH 10.00 buffers into the mini buffer bottles, taking care not to overfill.

      Standardize the pH meter using the three buffer solutions following the procedure outlined in the Appendix of this manual, pH Meter Operating Instructions.

      Always keep the pH electrode in the electrode storage solution when not in use.

2. Prepare your titrant.

    a. Read and understand how to fill and use a buret by reading the Common Laboratory Procedures section in the Appendix of this manual. Failure to properly fill the buret properly can result in spills and injuries.

    b. Condition a 25 mL buret with your standardized NaOH solution. Remember to check that the stopcock is closed before filling a buret. While holding the buret at a safe level over the sink, use a beaker to pour in your sodium hydroxide solution.

    c. After conditioning, fill the buret to above the zero mark with a beaker, dispel any air bubbles from the stopcock, and place the buret in the buret clamp. Record the initial buret reading to two decimal places, eg. 1.24 mL.

3. Prepare your analyte.

    a. Using a 10 mL volumetric pipet, accurately transfer 30.00 mL of 0.05 M acetic acid to a 150 mL beaker.

    b. To this solution, add 3–5 drops of thymolphthalein indicator and carefully place a clean magnetic stir bar into the beaker, without splashing.

4. Complete the titration set-up:

    a. Set up the stir plate underneath the buret containing the sodium hydroxide titrant.

    b. Place the beaker containing your dilute acetic acid solution onto the stir plate.

    c. Clamp the pH electrode in place below the level of the liquid in the beaker and away from the stir bar. Adjust the position of the buret tip so that it is inside the beaker, away from the side but with the stopcock at a convenient location for you to manipulate. Position the beaker so that the stir bar is somewhat displaced away from the center of the beaker to allow room for the pH electrode but make sure that the stir bar is above the center of the stir plate.

The Titration

5. First, you will perform a quick titration to find the approximate end point.

    To do so, you will add the titrant (0.2 M NaOH) in small increments, and recording the volume reading in the buret and the pH reading from the pH meter in your note book after each addition. Follow the instruction closely and add the titrant in the correct increments.

Lab Skill Tips

Read the buret at or slightly below eye level. Do not hold your hands or lab manual behind the buret.

You can hold a sheet of colored paper behind the buret to read the meniscus clearly.

Titration Tips

- **DO NOT** use your wash bottle to rinse down the sides of the beaker, as any added volume will change the pH readings and invalidate the titration curve data being collected.
- Record your buret readings after the addition of each increment.
- Allow time for the reaction vessel to become equilibrated and for the pH reading to become stabilized and then record the pH value in your notebook alongside the buret reading.
- When collecting data, leave an empty column between the buret reading and the pH in which to place the volume of NaOH added (difference between present buret reading and initial buret reading).

   a. Before adding any titrant, record the pH of the dilute acetic acid solution.

   b. Carefully add NaOH in 1 mL increments to the beaker until pH > 11. Record the buret reading and the pH reading. Note any color changes that occur alongside your buret and pH readings in your notebook.

   c. When you have completed this titration, transfer the solution in the titration flask to an 800 mL beaker.

6. Estimate the equivalence point and the midpoint by graphing pH vs. volume of NaOH added.

   a. Convert your buret readings to volumes of NaOH added.

   b. In your notebook, graph a titration curve by plotting the pH level on the **y-axis** and the volume of NaOH added on the **x-axis**.

   c. Find the area of the graph where the change in pH is the greatest, in other words, where the slope is the highest. The equivalence point is in this region.

   d. Consider the volumes of NaOH that bracket this region and estimate the volume of NaOH needed to reach the **equivalence** point.

   e. Estimate the volume of NaOH needed to reach the **midpoint** of the titration.

7. Refill your buret with the appropriate solutions and prepare another sample to be titrated following the same set up procedures as the first titration.

8. Perform a precise titration to accurately determine the midpoint and the equivalence point.

   To do so, you will add the titrant in **very** small increments before and after the estimated midpoint and equivalence point, while recording the

volume reading in the buret and the pH reading from the pH meter in your note book after each addition. Follow the instruction closely and add the titrant in the correct increments.

a. Before adding any titrant, record the pH of the dilute acetic acid solution.

b. Add 1 mL increments of NaOH until you are within **2 mL** of the midpoint. Record the buret volume reading and the pH reading after each addition.

c. Once you are within 2 mL of the estimated midpoint, add NaOH 2 drops at a time until you are 2 mL beyond the midpoint. Record the buret reading and the pH after each 2-drop addition.

d. Add 1 mL increments of NaOH until you are within 2 mL of the endpoint. Record the buret reading and the pH after each addition.

e. Once you are within 2 mL of the estimated end point, add NaOH 2 drops at a time until you are 2 mL beyond the estimated end point. Record the buret reading and the pH after each addition.

f. Add 1 mL increments of NaOH until pH > 11. Record the buret reading and the pH after each addition.

g. When you have completed this titration, pour the solution in the titration flask into an 800 mL beaker.

9. Perform another precise titration following the steps from 8a–g.

Clean Up

- Tightly cap and store the standardized NaOH and HCl solutions for later use.
- Adjust the pH of the used solution before disposal.
  - Drain the remaining NaOH from the buret into the 800 mL beaker. Slowly and carefully add any remaining 0.05 M acetic acid to the beaker.
  - Slowly and carefully, add 1 g of sodium bicarbonate to the solution in the 800 mL beaker. Pour this solution into the sink with copious amount of water.
- Rinse the buret and return it to the correct storage cabinet.
- Store the pH electrode in the appropriate storage solution container.

► **Continue to SAVE your standardized 0.2 M HCl and 0.2 M NaOH. You will use these solutions for the next 2 experiments.**

Data Analysis

Generating titration curves

1. Use a spreadsheet program such as Excel to enter the buret readings and corresponding pH for each titration. Use the buret readings to calculate the total volume of NaOH added at each point in the titration.

   - Tip: The total volume of NaOH added at any point in the titration can be calculated by taking the difference between that buret reading and the initial buret reading.

2. For each trial, generate a titration curve scatter plot of the total volume of NaOH added vs. pH. Make sure to include a plot title and labeled axes with units.

Generating 1st and 2nd derivative curves

1. As instructed in the "Strong Acid-Strong Base Titration" experiment, calculate "Vm" (midpoint volume), "D1" (1st forward divided difference), "Vd" (derivative midpoint volume), and "D2" (2nd forward divided difference) for each trial. Use these values to graph the approximations to the 1st and 2nd derivatives as you did in the previous laboratory. You may find it convenient to copy and modify the spreadsheet program you prepared to work up the data for that experiment and use it here.

   Check with your teaching assistant to see how they want the graphs turned in (i.e. by email or printed copies). Make sure your name is on each graph and that you have clearly titled and labeled the vertical and horizontal axes.

   If you completed 3 trials, you will only turn in graphs for the 2nd and 3rd trials (6 graphs total). You will still need to make graphs for the 1st trial to determine the endpoint and midpoint, but you **do not** need to turn in these graphs.

2. Using the derivative graphs, estimate the volume of NaOH required to reach the equivalence point for each of your trials. You should be able to make this estimate to within 0.02 mL, e.g. 10.98 mL.

Calculating molarity of acetic acid solution

1. Write down the chemical equation for the neutralization reaction of acetic acid with NaOH. Determine the stoichiometric ratio between these two reagents. In other words, how many moles of NaOH react with each mole of acetic acid?

2. Using the molarity of your standardized NaOH solution and the volume of NaOH dispensed at the equivalence point, determine how many moles of NaOH were dispensed in your first titration at the equivalence point.

3. Using the molar ratio of NaOH reacting with acetic acid, the moles of NaOH calculated in step 2, and the acetic acid sample volume, determine the molarity of your acetic acid solution. Remember to pay attention to units!

4. Repeat steps 1-3 to calculate the molarity of acetic acid for each titration.

Determining K_a of acetic acid prior to titration

1. Write down the chemical equation for the dissociation of acetic acid in water, which results in acetate ion and hydronium ion (H_3O^+).
   - Tip: equation (2) in the introduction is a generic version of this dissociation reaction.
2. Set up an "ICE" table for this reaction. "ICE" stands for "initial", "change", and "equilibrium" concentrations. Complete the table using the molarity of acetic acid solution in your first trial and a variable (i.e. "*x*") to represent change in concentration over the course of dissociation. Keep in mind that since water is a pure liquid, change in concentration is omitted from ICE tables A sample ICE table has been provided below.

	$HA(aq)$	+	$H_2O(l)$	⇌	$A^-(aq)$	+	H_3O^+
I	molarity		--		0		0
C	$-x$		--		$+x$		$+x$
E	molarity $- x$		--		x		x

3. Based on the dissociation chemical equation for acetic acid, write an equilibrium constant equation describing K_a using concentrations.
   - Tip: equation (3) in the introduction is a generic version of this K_a equation.
4. Insert the "equilibrium" concentration of each reagent from the ICE table into the K_a equation. The equation for K_a should now be written using the molarity of acetic acid solution and a variable (i.e. "*x*").
5. For your first trial, determine the concentration of H_3O^+ in the acetic acid solution using pH.
   - Tip: the concentration of H_3O^+ is equal to 10^{-pH}.
6. Using the equation for K_a, the molarity of acetic acid solution, and the concentration of H_3O^+, determine K_a of acetic acid prior to the titration.
7. Calculate K_a prior to titration for each trial, and then determine the average K_a of acetic acid.
8. Calculate the standard deviation of the average K_a prior to titration.
9. Calculate the 90% confidence limit for the average K_a prior to titration.

Determining K_a of acetic acid at the midpoint

The midpoint of a titration occurs when ½ of the original acid has reacted with the added strong base. This means ½ of the original acid remains in solution while ½ exists as the conjugate base. In other words, the number of moles of the acid, HA, remaining in solution is equal to the number of moles of conjugate base, A^-, and thus $[HA] = [A^-]$.

1. For your first trial, determine the volume of NaOH necessary to reach the midpoint. This should be equal to ½ of the volume required to reach the equivalence point.
2. Determine the pH of solution at the midpoint.
3. Examine the equilibrium constant equation for K_a for the dissociation of acetic acid. Given the assumption that $[HA] = [A^-]$ at the midpoint, are there any simplifications that can be made to this equation?
4. Using the midpoint pH, calculate the concentration of H_3O^+ at the midpoint.
5. Using the concentration of H_3O^+ at the midpoint and your simplified equation for K_a, determine K_a of acetic acid at the midpoint.
6. Calculate K_a at the midpoint for each trial, and then determine the average K_a of acetic acid.
7. Calculate the standard deviation of the average K_a at the midpoint.
8. Calculate the 90% confidence limit for the average K_a at the midpoint.

Determining K_a of acetic acid at the equivalence point

When determining K_a at the equivalence point, there are two chemical reactions that need to be considered: the reaction of acetic acid with sodium hydroxide, and the re-establishment of dissociation equilibrium between acetic acid and its conjugate base

1. Write down the chemical equation for the reaction of acetic acid with NaOH.
    - Tip: equation (7) in the introduction is a generic version of this reaction.
2. Set up and complete an "ICE" table for this reaction.
    - Tip: At the equivalence point, all moles of acetic acid have reacted with NaOH to become acetate ion.
3. Use the initial molarity of acetic acid, the equivalence point volume of NaOH, and the starting volume of acetic acid solution for your first trial to determine the concentration of acetate ion at the equivalence point.
4. Write down the chemical equation for the re-establishment of dissociation equilibrium between acetate ion and acetic acid.
    - Tip: equation (8) in the introduction is a generic version of this reaction.
5. Set up an "ICE" table for this reaction. Complete the table for each trial, using the molarity of acetate ion calculated above and a variable (i.e. "*x*") to represent change in concentration.

6. Based on the equation for re-establishment of dissociation equilibrium between acetate ion and acetic acid, write an equilibrium constant equation describing the **base dissociation constant, K_b.**
    - Tip: equation (9) in the introduction is a generic version of this K_b equation.
7. Insert the equilibrium concentration of each reagent from the re-establishment of equilibrium ICE table into the K_b equation. The equation for K_b should now be written using the acetate ion concentration and a variable (i.e. "*x*").
8. Determine the pOH of the solution at the equivalence point. pOH can be calculated using the equation pOH = 14 - pH.
9. Determine the concentration of OH^- in solution after the re-establishment of dissociation equilibrium between acetate ion and acetic acid. Similarly to how the concentration of H_3O^+ can be calculated from pH using 10^{-pH}, the concentration of OH^- can be calculated from pOH using 10^{-pOH}.
10. Using the equation for K_b, the molarity of acetate ion, and the concentration of OH-, determine K_b of the re-establishment of dissociation equilibrium at the equivalence point.
11. K_a can now be calculated using equation (10) from the introduction, $K_w = K_aK_b$.
12. Calculate K_a for each trial, and determine the average K_a of acetic acid at the equivalence point.
13. Calculate the standard deviation of the average K_a at the equivalence point.
14. Calculate the 90% confidence limit for the average K_a at the equivalence point.

Determining concentration of acetic acid and acetate ion at midpoint

1. Write down the chemical equation for the reaction of acetic acid with NaOH.
    - Tip: equation (7) in the introduction is a generic version of this reaction.
2. Set up and complete an "ICE" table for this reaction using the initial molarity of acetic acid.
    - Tip: Your equilibrium concentration for acetic acid and acetate ion should reflect that ½ of the acetic acid has been converted to acetate ion.
3. Use the initial concentration of acetic acid, the midpoint point volume of NaOH, and the starting volume of acetic acid solution to determine the concentration of both acetic acid and acetate ion at the midpoint.
4. Now we will consider how the re-establishment of dissociation equilibrium affects the concentration of acetic acid and acetate ion. Write down the chemical equation for the dissociation of acetic acid in water, which results in acetate ion and hydronium ion (H_3O^+).
5. Set up and complete an "ICE" table for this reaction using the molarity of acetic acid and acetate ion calculated above, and a variable (i.e. "*x*").
6. Determine the concentration of H_3O^+ in solution using the midpoint pH.

7. Apply the midpoint concentration of H_3O^+ to the equilibrium concentrations in the ICE table from step 5. Calculate the midpoint concentrations of acetic acid and acetate ion.

Polyprotic Systems

Introduction

Until now you have dealt primarily with monoprotic acids such as hydrochloric and nitric acid in the laboratory. This leaves an entire world of polyprotic acids unexplored. Polyprotic acids, acids that have more than one acidic proton, are common. For example, you have worked with sulfuric acid and with KHP that comes from diprotic phthalic acid. In this experiment, you will trace out the entire titration curve of the diprotic acid, carbonic acid H_2CO_3.

Carbonic acid in nature

The carbonic acid in our environment was formed by dissolving CO_2 from the air into water, or by water percolating through stone and rocks containing carbonate minerals. The carbonic acid system plays a major role in the respiration of all animals, including humans. The equilibrium among $CO_2(g)$, $H_2O(l)$, $HCO_3^-(aq)$, and $CO_3^{2-}(aq)$ is critical for the proper transport of CO_2, formed via cellular metabolism, through the bloodstream to be expelled by the lungs. While carbonic acid is not a strong acid by the dissociation definition, it is corrosive and does react with metals to form carbonates.

In this experiment we will start with Na_2CO_3, which dissociates into 2 Na^+ and carbonate, CO_3^{2-}, in water. We will then add acid, detecting the formation of each of the two endpoints of the titration curve using a pH meter. One aspect of polyprotic acids that is different from monoprotic acids is that they always make buffer solutions. Think about your list of strong acids: all except sulfuric acid are monoprotic acids, and only the first proton of sulfuric acid is considered strong. This buffering action can make experiments more complicated. In the experiment you are about to perform, titration of the first endpoint that you encounter establishes a buffer solution that complicates the analysis and determination of K_a for that equivalence point. We should note here that this buffering action can also be used to your benefit. Some reactions take place only in a specific pH range, and buffers can be used to maintain this pH during an experiment. You will be examining the nature of buffer solutions in the next experiment in the series on acid-base chemistry.

Polyprotic acids can generate very complex systems at equilibrium, and can undergo multiple separate dissociations. Carbonic acid undergoes two separate dissociations:

$$H_2CO_3(aq) \rightleftharpoons H^+(aq) + HCO_3^-(aq) \qquad K_{a1} = 4.4 \times 10^{-7}$$

$$HCO_3^-(aq) \rightleftharpoons H^+(aq) + CO_3^{2-}(aq) \qquad K_{a2} = 4.7 \times 10^{-11}$$

Each of these dissociations is an equilibrium reaction with an acid dissociation constant. As a result, calculating the concentrations of the species present in solution can become quite involved.

$$H^+(aq) + CO_3^{2-}(aq) \rightleftharpoons HCO_3^-(aq) \qquad K_{a2} = 4.7 \times 10^{-11}$$

$$H^+(aq) + HCO_3^-(aq) \rightleftharpoons H_2CO_3(aq) \rightarrow H_2O(l) + CO_2(g) \qquad K_{a1} = 4.4 \times 10^{-7}$$

We can also write the acid dissociation reactions of carbonic acid in reverse of the usual direction to emphasize that we are starting from a solution of Na_2CO_3. Notice that during the titration you will encounter the equivalence point of the **second** proton (K_{a2}) of diprotic carbonic acid as the first observed equivalence point in the titration. It occurs at high pH. The **first** proton (K_{a1}) is encountered as the second observed equivalence point in the titration. It occurs at low pH. One of the goals of this experiment will be to make your own determinations of the two acid dissociation constants of carbonic acid.

Because of the polyprotic nature of carbonic acid, the equilibrium analysis necessary to develop the formulas for reduction of measurements of the pH into the acid dissociation constant are somewhat involved for the second proton equilibrium (the first observed equivalence point that you will encounter in the titration). We will not go through the details of the development, but will just describe for you how to find the final formulas. You may want to go through the development on your own, using the discussion as an aid to prove to yourself that the formulas are correct. At the second proton equivalence point, the solution is identical in composition with a solution of the sodium salt of the bicarbonate ion HCO_3^- (except for some extra dissolved NaCl*(aq)*). An equilibrium treatment of the pH of that solution will yield precisely the formulas we need to work with. The dominant species equilibria to be considered are:

$$HCO_3^-(aq) + H_2O(l) \rightleftharpoons H_2CO_3(aq) + OH^-(aq) \qquad K_{b2} = K_w/K_{a1}$$

$$HCO_3^-(aq) + H_2O(l) \rightleftharpoons H_3O^+(aq) + CO_3^{2-}(aq) \qquad K_{a2}$$

We start by writing down the two conditions that are commonly referred to as a mass balance and a charge balance. The mass balance sets the sum of all carbonate containing species equal to the total concentration in the original sample as diluted to the present volume. The charge balance sets the sum of the concentrations of all positively charged species equal to the sum of the concentrations of all the negatively charged species (including the sodium cation needed for $NaHCO_3$). These two conditions are combined into an equality that must be observed.

We then use the two equilibrium expressions listed above and the K_w equilibrium to re-express $[H_2CO_3]$, $[CO_3^{2-}]$, and $[OH^-]$, and insert these into the combined equality. The combined equality is then simplified and rearranged to get the result:

$$[H_3O^+] = \sqrt{\frac{K_{a2}[HCO_3^-] + K_w}{1 + \frac{[HCO_3^-]}{K_{a1}}}}$$

While this formula looks difficult to work with, the specific circumstances of the carbonic acid equivalence point simplify it greatly. Firstly, for convenient laboratory concentrations, and specifically, for those used in this experiment, it will be true that $[HCO_3^-] >> K_{a1}$. Consequently, we may neglect the unity in the denominator.

Further, it will also be the case that $K_{a2}[HCO_3^-] >> K_w$, so that K_w may be neglected in the numerator. Canceling and simplifying then gives:

$$[H_3O^+] = \sqrt{K_{a1}K_{a2}} \qquad (1)$$

While this does not give us either of the acid constants directly, if we know one of them, we can use this relationship to determine the other.

From the equilibrium at the second observed equivalence point we get the necessary additional information that enables the determination of both acid dissociation constants. At the second observed equivalence point, the solution has had two equivalents of protons added to the analyte. For purposes of consideration of the pH equilibria, the solution is then simply that of carbonic acid H_2CO_3 (with some extra NaCl in solution that does not affect the acid equilibria).

$$H_2CO_3(aq) \rightleftharpoons H^+(aq) + HCO_3^-(aq) \qquad K_{a1}$$

$$HCO_3^-(aq) \rightleftharpoons H^+(aq) + CO_3^{2-}(aq) \qquad K_{a2}$$

A fairly quick solution of these equilibria is available if $K_{a1} >> K_{a2}$ because then we may assume that the $[H^+]$ concentration arises dominantly from the first equilibrium, and then $[H^+] = [HCO_3^-]$.

Writing the equilibrium constant expressions:

$$K_{a1} = \frac{[H^+][HCO_3^-]}{[H_2CO_3]};\ K_{a2} = \frac{[H^+][CO_3^{2-}]}{[HCO_3^-]}$$

Rearranging these expressions:

$$[H_2CO_3] = \frac{[H^+][HCO_3^-]}{K_{a1}}, [HCO_3^-] = \frac{[H^+][CO_3^{2-}]}{K_{a2}}$$

Using substitution:

$$[H_2CO_3] = \frac{[H^+]^2[CO_3^{2-}]}{K_{a1}K_{a2}}$$

Since $[H^+] = [HCO_3^-]$, solving for $[CO_3^{2-}]$ in the previous expression gives:

$$[CO_3^{2-}] = \frac{[HCO_3^-]}{[H^+]}K_{a2} = K_{a2}$$

This reduces the expression for $[H_2CO_3]$ to:

$$[H_2CO_3] = \frac{[H^+]^2}{K_{a1}}$$

Now in a solution that is M molar in H_2CO_3, we must have:

$$[HCO_3^-] + [CO_3^{2-}] + [H_2CO_3] = M$$

$$[H_2CO_3] = M - ([HCO_3^-] + [CO_3^{2-}])$$

Since we are dealing with weak acid dissociation constants, we can expect

$$[HCO_3^-] + [CO_3^{2-}] << M$$

$$\text{hence } [H_2CO_3] = M$$

Using the concentrations in the expression for K_{a1},

$$K_{a1} = \frac{[H^+]^2}{M} \qquad (2)$$
$$[H^+] = \sqrt{MK_{a1}}$$

In the titration, $M = a/(V + v)$, where $a = g/(105.99\ g/mol)$, the number of moles of sodium carbonate in the sample, g = grams of Na_2CO_3 in the titrated sample, V is the original volume of water in which the sample was dissolved, and v is the volume of HCl added to reach the second observed equivalence point in the titration. Of course in both equations (1) and (2), $[H^+] = \text{antilog}_{10}(-\text{pH})$. Once K_{a1} is found, equation (1) may be used to find K_{a2}.

In preparation for the Acid-Base Buffer experiment, obtain your group number for your assigned pH values from your TA.

Write your Group Number here: _____

Learning Goals

The following is a list of skills that you will use in this experiment.

Laboratory	• Calculating volume and mass of reagents used to prepare solutions • Performing a titration
Conceptual	• Properties of polyprotic acids (i.e. multiple dissociations)
Data Analysis	• Properties of a polyprotic acid titration curve • Calculating acid-base dissociation constants from $[H_3O^+]$

Procedure

Work in pairs on this experiment.

Each student must collect data and submit a separate report. The actual data analyses and the written reports must be done entirely independently of your lab partner or other students. Make sure that you avoid unauthorized collaboration and plagiarism. All suspected violations of the Code of Academic Conduct will be referred to Student Judicial Affairs.

Stock Chemicals Used

Chemical	Maximum Amount Used
0.04% Bromocresol Green Indicator	Drops
1% Phenolphthalein Indicator	Drops
pH Meter Calibration Buffer, pH 4 (red)	< 5 mL
pH Meter Calibration Buffer, pH 7 (yellow)	< 5 mL
pH Meter Calibration Buffer, pH 10 (blue)	< 5 mL
Sodium Carbonate, (s)	< 1 g

Safety First

Remember to always wear gloves and take caution when handling all acids and bases.

Wear your goggles!

Titration Set Up

1. Prepare the sodium carbonate solution.
    a. For this procedure, you will need dried sodium carbonate, which you have prepared and stored in a desiccator during the previous experiment.
    b. Accurately weigh one sample of 0.10–0.15 g sodium carbonate by difference into a 150 mL beaker. Record the mass in your notebook.
    c. Add 30 mL of precisely measured DI water to the titration vessel. Record the volume added to the nearest 0.02 mL. Make sure the sodium carbonate is **fully dissolved** before starting the titrations.
2. Prepare the titrant.
    a. Find your 1 L bottle of your standardized HCl from the previous experiments. Before opening the bottle, carefully invert it several times to ensure that your solution is uniform.
    b. Condition a 25 mL buret with your standardized HCl solution. Remember to check that the stopcock is closed before filling a buret. While holding the buret at a safe level, use a beaker to pour in your hydrochloric acid solution.

Safety First

Before inverting the HCl bottle, make sure it is properly capped.

Lab Skill Tips

Read the buret at or slightly below eye level. Do not hold your hands or lab manual behind the buret.

You can hold a sheet of colored paper behind the buret to read the meniscus clearly.

c. After conditioning, fill the buret to above the zero mark with a beaker, place the buret in the clamp, and dispel any air bubbles from the stopcock. Record the initial buret reading to two decimal places, e.g. 1.24 mL. **Remember to check that the stopcock is closed before filling the buret.**

Tips on using a buret

- Be careful when filling the buret. Only one person should be filling the buret. Be sure the stopcock is closed before filling.
- Use a 100 or 150mL beaker to fill the buret. Never use flasks, 1L plastic bottles, or large beakers to fill the buret.

3. Calibrate the pH meter.

   a. The pH meter will need to be calibrated before starting the experiment. There is no need to recalibrate later during the experiment.

   b. Dispense the pH 4.00, pH 7.00, and pH 10.00 buffers into the mini buffer bottles, taking care not to overfill. Standardize the pH meter using the three buffer solutions following the procedure outlined in the Appendix of this manual, pH Meter Operating Instructions.

Care of pH meter and pH electrodes

- Keep the tip of the electrode submerged in solution at all times.
- Never leave the electrode in deionized water.
- When rinsing the electrode use a light stream of deionized water.
- Be careful of the electrode when adding strong base or when stirring the solution.
- After completing the experiment, STORE THE ELECTRODE IN THE STORAGE SOLUTION provided. Additional storage solution is available in the laboratory if needed.

4. Complete the titration set-up:

   a. Set up the stir plate underneath the buret containing the HCl titrant.

   b. Place the titration vessel with your sodium carbonate solution onto the stir plate.

   c. To the titration vessel, add 3–5 drops of phenolphthalein indicator.

   d. Clamp the pH electrode in place below the level of the liquid in the beaker and away from the stir bar. Adjust the position of the buret tip so that it is inside the vessel, away from the side but with the stopcock at a convenient location for you to manipulate. Position the titration vessel so that the stir bar is somewhat displaced away

from the center to allow room for the pH electrode but make sure that the stir bar is above the center of the stir plate.

The Titrations

5. Perform the first titration.

   To do so, you will add the titrant (0.2 M HCl) in small increments throughout the titration, and in very small increments before and after the estimated equivalence points. Follow the instruction closely and add the titrant in the correct increments. Record all relevant observations in your notebook.

   Work efficiently by having one partner add the titrant and report the buret reading while the other read the pH meter and record the data. Make sure that each partner has a complete set of data after a complete titration is finished. Make sure the data for the sodium carbonate (including the mass of the sodium carbonate and volume of water added) are clearly recorded in your notebook.

Titration Tips

- **DO NOT** use your wash bottle to rinse down the sides of the beaker, as any added volume will change the pH readings and invalidate the titration curve data being collected.
- Record your buret readings after the addition of each increment.
- Allow time for the reaction vessel to become equilibrated and for the pH reading to become stabilized and then record the pH value in your notebook alongside the buret reading.
- When collecting data, leave an empty column between the buret reading and the pH in which to place the volume of NaOH added (difference between present buret reading and initial buret reading).

   a. Turn on the magnetic stir bar slowly and increase the setting gradually until you have it rotating at a moderate speed.
   b. Before adding any titrant, record the buret reading the pH of the analyte solution.
   c. Add HCl in 1 mL increments until the pH reaches 9.6. Record the buret volume reading and the pH reading after each addition.
   d. Once the pH reaches 9.6, add HCl in 0.10 mL increments until a **pH of 7 or lower** is reached. Record the buret reading and the pH after each addition.
      i. The solution should turn clear within this pH interval. Record any color change you observe next to the corresponding pH reading.

ii. After the solution turns clear, add 3–5 drops of bromocresol green indicator.

iii. Continue to add HCl in 0.10 mL increments until you have added a total of 1 mL of titrant **past the volume at which the solution turned clear.** Record the buret volume reading and the pH reading after each addition.

e. Add HCl in 1 mL increments until the pH reaches 5.5. Record the buret volume reading and the pH reading after each addition.

f. Once pH is reaches 5.5, add HCl in 0.10 mL increments until you observe a color change. Record the buret reading and the pH after each addition. Record any color change you observe next to the pH reading.

g. After you observe the color change, continue adding HCl in 0.10 mL increments until you have added a total of 2 mL of titrant after the color change. Record the buret volume reading and the pH reading after each addition.

h. Add HCl in 1 mL increments until you have added a total of 3 mL after the last step. Record the buret volume reading and the pH reading after each addition.

i. When you have completed this titration, pour the solution in the titration vessel into an 800 mL beaker.

Lab Skill Tip

You do not need to calibrate the pH meter again after each titration.

6. You will now repeat the titration for the remaining two samples by repeating steps 1–5.

   a. Exchange roles if you did not trade off during the first titration. Each partner needs to have performed all roles.

   b. You should modify your technique for the remaining two samples based on your experience with the first one. You may find these general directions need to be slightly adjusted to improve the quality of data for your curve, for example by choosing a somewhat different specific pH at which to change the increment sizes.

Clean Up

- Tightly cap and store the bottles containing the standardized NaOH and HCl solutions for use in later experiments.
- Adjust the pH of the used solution before disposal.
  - Drain the remaining HCl from the buret into the 800 mL beaker.
  - Slowly and carefully, add your remaining sodium carbonate sample into the 800 mL beaker. Pour this solution into the sink with copious amount of water.
- Rinse the buret and return it to the correct storage cabinet.
- Store the pH electrode in the appropriate storage solution container.

► **Continue to SAVE your standardized 0.2 M HCl and 0.2 M NaOH. You will use these solutions for the next experiment.**

Data Analysis

Generating titration curves

1. Use a spreadsheet program such as Excel to enter the buret readings and corresponding pH for each titration. Use the buret readings to calculate the total volume of HCl added at each point in the titration.

   - Tip: The total volume of HCl added at any point in the titration can be calculated by taking the difference between that buret reading and the initial buret reading.

2. For each trial, generate a titration curve scatter plot of the total volume of HCl added vs. pH. Make sure to include a plot title and labeled axes with units.

Generating 1st and 2nd derivative curves

1. As instructed in the "Strong Acid-Strong Base Titration" experiment, calculate "Vm" (midpoint volume), "D1" (1st forward divided difference), "Vd" (derivative midpoint volume), and "D2" (2nd forward divided difference) for each trial. Use these values to graph the approximations to the 1st and 2nd derivatives as you did in the previous laboratory. You may find it convenient to copy and modify the spreadsheet program you prepared to work up the data for that experiment and use it here.

   Check with your teaching assistant to see how they want the graphs turned in (i.e. by email or printed copies). Make sure your name is on each graph and that you have clearly titled and labeled the vertical and horizontal axes.

   If you completed 3 trials, you will only turn in graphs for the 2nd and 3rd trials (6 graphs total). You will still need to make graphs for the 1st trial to determine the endpoint and midpoint, but you **do not** need to turn in these graphs.

2. Using the derivative graphs, estimate the volume of HCl required to reach the **2** equivalence points for each of your trials. You should be able to make this estimate to within 0.02 mL, e.g. 10.98 mL.

Calculating K_{a1} from the second observed equivalence point

As you calculate K_{a1} and K_{a2}, keep in mind this titration was performed in "reverse". Instead of using a base to *remove* protons, as was observed in previous labs, HCl was used to *add* protons to CO_3^{2-} one at a time. The second proton was added first, at the first observed equivalence point. The first proton was added at the second observed equivalence point. Therefore the dissociation constant for the first proton, K_{a1}, is calculated using data from the second observed equivalence point.

1. For your first trial, what is the pH at the **second observed equivalence point**? Use this value to determine the concentration of H^+.

2. Using the mass of Na_2CO_3 from your first trial, and the molar mass of Na_2CO_3, determine how many moles of carbonate species are present in the sample.

3. Use the moles of carbonate species calculated above, the volume of water used to prepare your solution, and the volume of HCl added at the **second observed equivalence point** to determine the molarity of carbonate species in solution.

4. Following equation (2) in the introduction, determine the dissociation constant for the first proton, K_{a1}, using the concentration of H^+ and the molarity of carbonate species in solution.

5. Calculate K_{a1} for each trial, and determine the average K_{a1}.

6. Calculate the standard deviation of the average K_{a1}.

7. Calculate the 90% confidence limit for the average K_{a1}.

Calculating K_{a2} from the first observed equivalence point

Now that you have determined the acid dissociation constant for the first, most acidic proton, K_{a1}, you can determine the acid dissociation constant for the second, less acidic proton, K_{a2}. As a reminder, K_{a2} will be found using data from the first observed equivalence point.

1. For your first trial, what is the pH at the **first observed equivalence point**? Use this value to determine the concentration of H^+.

2. Following equation (1) in the introduction, determine the dissociation constant for the second proton, K_{a2}, using the concentration of H^+ at the first observed equivalence point and the dissociation constant for the first proton, K_{a1}. For our purposes, $[H^+]$ is equivalent to $[H_3O^+]$.

3. Calculate K_{a2} for each trial, and determine the average K_{a2}.

4. Calculate the standard deviation of the average K_{a2}.

5. Calculate the 90% confidence limit for the average K_{a2}.

Acid-Base Buffers

Introduction

In this experiment we will focus on the topic of acid-base buffers. An acid-base buffer is a solution that resists pH change. Buffers are very important in chemistry since many reactions will only occur in certain pH ranges. This is especially true of many biological systems in which the pH must be maintained in very narrow ranges if the organism is to survive.

Buffers are solutions that simultaneously contain relatively large amounts of acid/base conjugate pairs. An example that you are already familiar with is the acetic acid/acetate ion conjugate pair. A solution containing both of these substances will be a buffer because the weak acid will react with added base to produce the conjugate base via:

$$CH_3COOH(aq) + OH^-(aq) \rightleftharpoons CH_3COO^-(aq) + H_2O(l)$$

and the conjugate base present will react with added acid to produce the conjugate acid via:

$$CH_3COO^-(aq) + H_3O^+(aq) \rightleftharpoons CH_3COOH(aq) + H_2O(l)$$

In both cases the pH will change with the addition of acid or base, however the pH will change very little if the amounts of added base or acid is small relative to the concentration of the buffer conjugates already present in the solution.

Additionally, a buffer works best when the pH is about the same as the pK_a for the acid component of the buffer. To illustrate this, consider the reaction:

$$HA(aq) + H_2O(l) \rightleftharpoons A^-(aq) + H_3O^+(aq)$$

for which the K_a expression is:

$$K_a = \frac{[H_3O^+][A^-]}{[HA]}$$

If we take the –log of both sides then we have,

$$-\log K_a = -\log[H_3O^+] - \log\frac{[A^-]}{[HA]}$$

or

$$pH = pK_a + \log\frac{[A^-]}{[HA]}$$

Considering the second term in the above equation, we see that in order for the pH change to be minimal, the contribution of the logarithm must be small. In fact, the logarithm will be zero if $[A^-] = [HA]$ since $\log 1 = 0$. Therefore, as strong acids or bases are added, we can expect a buffer solution to work best at stabilizing the pH when $[A^-] = [HA]$. If the pH is the same as the pK_a, it follows that $[A^-] = [HA]$. The above equation, called the **Henderson-Hasselbalch** equation, can also be used to determine the conjugate acid-base concentrations required to make a buffer of specified pH. We can rearrange this equation to express the conjugate acid-base concentration ratio in terms of pH. We do this by subtracting pK_a from both sides of the equation then taking the antilog of both sides. Recall that the antilog function is 10^x.

$$\frac{[A^-]}{[HA]} = 10^{(pH - pK_a)}$$

Given a target pH for the buffer and a desired concentration for either the conjugate acid or base, one can then find the concentration and thus a mass or volume required of the unspecified conjugate to complete the buffer solution.

Table 1 contains a list of useful pK_a values needed for this lab.

Table 1. pK_a values for Acids used in the experiment

Name of Acid	Dissociation Reaction	pKa
Acetic acid	$CH_3COOH(aq) + H_2O(l) \rightleftharpoons H_3O^+(aq) + CH_3COO^-(aq)$	4.74
Hydrogen carbonate ion	$HCO_3^-(aq) + H_2O(l) \rightleftharpoons H_3O^+(aq) + CO_3^{2-}(aq)$	10.33

In this experiment, you will prepare two buffers and study the effects of adding acid and base. For each of the buffers you will calculate the amounts of the conjugates required to prepare the buffer solutions. Then you will make small additions of acid and base to the buffer solutions and observe the pH changes that occur. You will graph these pH changes against volume and make comparisons to the previous experiments.

Enzymes, the Goldilocks of chemical reactions

Enzymes play a critical role in catalyzing chemical reactions within living organisms. The activity and function of an enzyme depends on its amino acid sequence. Intermolecular interactions between amino acids are affected by whether they are protonated or deprotonated, which in turn is affected by the pH of the surrounding environment. Enzymes can lose function or denature completely if their surroundings are too acidic or basic, so buffers play an essential role in maintaining environmental pH. For example, the cytoplasm within our cells contains a buffering system consisting of monohydrogen phosphate (HPO_4^{2-}) and dihydrogen phosphate ($H_2PO_4^-$). Monohydrogen phosphate has a pK_a of 6.8, which makes it a good buffer for maintaining an intracellular pH of about 7, the ideal pH for many enzymes in the human body.

Pre-Lab Preparation

The calculations for this experiment are not trivial. For this reason, you are required to prepare for this experiment by calculating the needed amounts of your reagents to make your buffer solutions at the assigned pH values. The Data Analysis section of this experiment contains questions to help guide your calculations.

You should have been assigned a group number during the previous laboratory session. (You were asked to write it down in your lab manual immediately following the introduction of the Polyprotic System Experiment.)

Table 2 identifies the assigned pH values by group numbers. If you do not complete the calculations before the laboratory session, you may not have time to complete this experiment.

Table 2. pH of Buffer Solutions

	Acidic Buffer	Basic Buffer
Group 1	4.5	10.0
Group 2	4.6	10.1
Group 3	4.7	10.2
Group 4	4.8	10.3
Group 5	4.9	10.4
Group 6	5.0	10.5

You must have the calculations checked by the teaching assistant before you can begin the laboratory experiment.

Hint

Using $M_1V_1=M_2V_2$, calculate the volume of 2.5 M sodium acetate solution needed to prepare your dilute sodium acetate solution.

Learning Goals

The following is a list of skills that you will use in this experiment.

Laboratory	• Using the Henderson-Hasselbalch equation to calculate buffered solutions of a specified pH
Conceptual	• Properties of buffers (i.e. resistance to pH change, presence of conjugate pairs)
Data Analysis	• Generating and analyzing titration curves of a buffered solution

Procedure

Work in pairs for this experiment.

Each student must collect data and submit a separate report. The actual data analyses and the written reports must be done entirely independently of your lab partners or other students. Make sure that you avoid unauthorized collaboration and plagiarism. All suspected violations of the Code of Academic Conduct will be referred to Student Judicial Affairs.

Stock Chemicals Used

Chemical	Maximum Amount Used
6 M Hydrochloric Acid	According to calculation (< 10 mL)
6 M Sodium Hydroxide	According to calculation (< 10 mL)
6 M Acetic Acid (CH_3COOH)	According to calculation (< 30 mL)
2.5 M Sodium Acetate ($NaCH_3COO{\cdot}3H_2O$)	According to calculation (< 25mL)
Sodium Carbonate (Na_2CO_3)	According to calculation (< 5 g)
Sodium Bicarbonate ($NaHCO_3$)	According to calculation (< 5 g)
pH Meter Calibration Buffer, pH 4 (red)	< 5 mL
pH Meter Calibration Buffer, pH 7 (yellow)	< 5 mL
pH Meter Calibration Buffer, pH 10 (blue)	< 5 mL

Safety First

Remember to always wear gloves and take caution when handling all acids and bases.

Wear your goggles!

Part I. Preparing Your Buffers

You will be cooperating with another group of students to prepare two 250 mL buffer solutions, an acidic and a basic buffer. Divide the tasks amongst yourselves so that each group receives approximately 120 mL of the correct acidic buffer and 120 mL of the basic buffer at the end of Part I.

The acidic buffer will be prepared from a 6 M acetic acid (CH_3COOH) solution and 2.5 M sodium acetate trihydrate ($NaCH_3COO{\cdot}3H_2O$). The basic buffer solution is prepared from solid sodium hydrogen carbonate ($NaHCO_3$) and solid anhydrous sodium carbonate (Na_2CO_3). After preparing the solutions you will measure the pH level of the solution and adjust the levels by adding either strong acid or strong base as needed.

1. **Group Member Assignments:** During the previous laboratory experiment, the TA assigned your group a number between **1 and 6**. Work with the other group with the **same buffer assignment** to prepare the 2 buffer solutions at the designated pH values given in Table 2.

   Have one group prepare the 250 mL of the acidic buffer and the other group prepare 250 mL of the basic buffer at the designated pH values.

2. Preparation of Acidic Buffer in a 250 mL volumetric flask:

   a. Calculate the volume of 2.5 M sodium acetate solution needed to prepare 250 mL of 0.10 M sodium acetate. DO NOT USE the 1.0 M sodium acetate.

   b. First, transfer approximately 15 mL of the 2.5 M sodium acetate solution into a small beaker, and then use a volumetric pipet to transfer the appropriate volume—calculated from the previous step—to your 250 mL volumetric flask. Add 120–150 mL of deionized water to the 250 volumetric flask and mix.

   c. Calculate the volume of 6 M stock acetic acid solution you need to make 250 mL of the buffer solution to the designated pH.

   d. Use your graduated cylinder to transfer this volume of 6 M acetic acid to the volumetric flask. Using a water bottle, fill the flask to the mark with deionized water to make a solution totaling 250 mL.

   e. Mix the solution well, then transfer the solution to an appropriately 400 mL beaker.

3. Preparation of Basic Buffer in a 250 mL volumetric flask:

   a. Calculate the mass of solid sodium hydrogen carbonate needed to prepare 250 mL of 0.050 M sodium hydrogen carbonate solution.

   b. Weigh out this mass of solid sodium hydrogen carbonate and quantitatively transfer it to a clean 250 mL volumetric flask. Add 120–150 mL of deionized water to the 250 volumetric flask and mix.

   c. Calculate the mass of solid anhydrous sodium carbonate required to make the basic buffer to the designated pH.

   d. Weigh out this mass of anhydrous sodium carbonate and quantitatively transfer it to your 250 mL volumetric flask. Now, fill your flask to the mark with deionized water to make a solution totaling 250 mL. After you have mixed the buffer solution well, place the buffer solution into a clean and appropriately labeled 400 mL beaker.

4. You will need to calibrate the pH meter at this point.

   a. The pH meter will need to be calibrated before starting the experiment. There is no need to recalibrate later during the experiment.

   b. Dispense the pH 4.00, pH 7.00, and pH 10.00 buffers into the mini buffer bottles, taking care not to overfill. Standardize the pH meter using the three buffer solutions following the procedure outlined in the Appendix of this manual, pH Meter Operating Instructions.

Care of pH meter and pH electrodes

- Keep the tip of the electrode submerged in solution at all times.
- Never leave the electrode in deionized water.
- When rinsing the electrode use a light stream of deionized water.
- Be careful of the electrode when adding strong base or when stirring the solution.
- After completing the experiment, **store the electrode in the storage solution provided.** Additional storage solution is available in the laboratory if needed.

5. Adjusting the pH of your buffer.

   Your buffer solution may not be at the assigned pH. Use drop-wise addition of 6 M HCl or 6 M NaOH to adjust the pH.

   a. Measure the pH of your buffer.

   b. Using a disposable pipet, add 6 M HCl drop-wise to lower the pH, or add 6 M NaOH drop-wise to raise the pH.

   c. Adjust the pH until it is equal to the assigned pH. Stir the solution and record the pH to the nearest 0.02 pH unit.

   d. Split the buffer between the groups. Carefully pour the buffers into four labeled 250 mL Erlenmeyer flasks. Make sure that each group has at least **80 mL** of the acidic or basic buffer.

Part II. Preparing Your Reagents

Safety First

Before inverting the HCl and NaOH bottles, make sure they are properly capped.

1. Find your 1 L bottle of your standardized HCl from the previous experiments. Before opening the bottle, carefully invert it several times to ensure that your solution is uniform.
2. Find your 1 L bottle of your standardized NaOH from the previous experiments. Before opening the bottle, carefully invert it several times to ensure that your solution is uniform.
3. Prepare 120 mL of 0.1 M acetic acid from the 6 M acetic acid stock solution using a 150 mL beaker. Once the solution has been made, transfer it to a 125 mL, 250 mL, or 500 mL Erlenmeyer flask for storage and label the Erlenmeyer flask with a graphite pencil.
4. Condition and fill two 25 mL burets, one with the 0.2 M HCl solution and the other buret with 0.2 M NaOH. Record the initial volume of HCl and NaOH to the nearest 0.02 mL.

Lab Skill Tips

Read the buret at or slightly below eye level. Do not hold your hands or lab manual behind the buret.

You can hold a sheet of colored paper behind the buret to read the meniscus clearly.

Tips on using a buret

- Be careful when filling the buret. Only one person should be filling the buret. Be sure the stopcock is closed before filling.
- Use a 100 or 150mL beaker to fill the buret. Never use flasks, 1L plastic bottles, or large beakers to fill the buret.

Part III. Titration with 0.2 M HCl

In this part of the experiment, each group will treat each of the two buffer solutions with 0.2 M HCl solution. You will add the titrant (0.2 M HCl) in small increments throughout the titration. You will then plot the pH vs. added volume of HCl to graphically observe the pH changes that occur. You will also explore the effect of adding 0.2 M HCl to 0.1 M acetic acid solution.

Titration Tips

- **DO NOT** use your wash bottle to rinse down the sides of the beaker, as any added volume will change the pH readings and invalidate the titration curve data being collected.
- Record your buret readings after the addition of each increment.
- Allow time for the reaction vessel to become equilibrated and for the pH reading to become stabilized and then record the pH value in your notebook alongside the buret reading.

1. Titrate the acidic buffer with 0.2 M HCl.
    a. Place 40 mL of the acidic buffer in a clean 150 mL beaker. Gently place a stir bar into the beaker.
    b. Set up the beaker containing the buffer, stir plate, and pH electrode under the buret containing the 0.2 M HCl solution. Start gently rotating the stir bar. Record the initial buret reading and pH meter reading.
    c. Add HCl in 1 mL increments to the buffer until the pH of the buffer has decreased by 1.5 pH units. Record the buret reading and the pH reading after each addition.
    d. **Save this solution** in an Erlenmeyer flask for use in the last part of this experiment.

2. Titrate the basic buffer with 0.2 M HCl.

    a. Place 40 mL of the basic buffer in a clean 150 mL beaker. Gently place a stir bar into the beaker.

    b. Set up the beaker containing the buffer, stir plate, and pH electrode under the buret containing the 0.2 M HCl solution. Start gently rotating the stir bar. Record the initial buret reading and pH meter reading.

    c. Add HCl in 1 mL increments to the buffer until the pH of the buffer has decreased by 1.5 pH units. Record the buret reading and the pH reading after each addition.

    d. When you have completed this titration, pour the solution in the titration vessel into an 800 mL beaker.

3. Titrate the 0.1 M acetic acid with 0.2 M HCl.

    a. Place 40 mL of the 0.1 M acetic acid in a clean 150 mL beaker. Gently place a stir bar into the beaker.

    b. Set up the beaker containing the acetic acid solution, stir plate, and pH electrode under the buret containing the 0.2 M HCl solution. Start gently rotating the stir bar. Record the initial buret reading and pH meter reading.

    c. Add HCl in 1 mL increments to the buffer until the pH of the solution has decreased by 1.5 pH units. Record the buret reading and the pH reading after each addition.

    d. When you have completed this titration, pour the solution in the titration vessel into an 800 mL beaker.

Part IV. Titration with 0.2 M NaOH

In this part of the experiment each group will treat each of the two buffer solutions with 0.2 M NaOH solution. You will add the titrant (0.2 M NaOH) in small increments throughout the titration. You will then plot the pH vs. added volume of NaOH to graphically observe the pH changes that occur. You will also explore the effect of adding 0.2 M NaOH to 0.1 M acetic acid solution.

1. Titrate the acidic buffer with 0.2 M NaOH.
    a. Place 40 mL of the acidic buffer in a clean 150 mL beaker. Gently place a stir bar into the beaker.
    b. Set up the beaker containing the buffer, stir plate, and pH electrode under the buret containing the 0.2 M NaOH solution. Start gently rotating the stir bar. Record the initial buret reading and pH meter reading.
    c. Add NaOH in 1 mL increments to the buffer until the pH of the buffer has increased by 1.5 pH units. Record the buret reading and the pH reading after each addition.
    d. When you have completed this titration, pour the solution in the titration vessel into an 800 mL beaker.
2. Titrate the basic buffer with 0.2 M NaOH.
    a. Place 40 mL of the basic buffer in a clean 150 mL beaker. Gently place a stir bar into the beaker.
    b. Set up the beaker containing the buffer, stir plate, and pH electrode under the buret containing the 0.2 M NaOH solution. Start gently rotating the stir bar. Record the initial buret reading and pH meter reading.
    c. Add NaOH in 1 mL increments to the buffer until the pH of the buffer has increased by 1.5 pH units. Record the buret reading and the pH reading after each addition.
    d. When you have completed this titration, pour the solution in the titration vessel into an 800 mL beaker.

3. Titrate the 0.1 M acetic acid with 0.2 M NaOH.

   a. Place 40 mL of the 0.1 M acetic acid in a clean 150 mL beaker. Gently place a stir bar into the beaker.

   b. Set up the beaker containing the acetic acid solution, stir plate, and electrode under the buret containing the 0.2 M NaOH solution. Start gently rotating the stir bar. Record the initial buret reading and pH meter reading.

   c. Add NaOH in 1 mL increments to the buffer until the pH of the solution has increased by 1.5 pH units. Record the buret reading and the pH reading after each addition.

   d. When you have completed this titration, pour the solution in the titration vessel into an 800 mL beaker.

4. Titrate your acetic acid buffer/HCl solution with 0.2 M NaOH.

   a. Transfer the acidic buffer/HCl mixture from Part III step 1 to a clean 150 mL beaker. Gently place a stir bar into the beaker.

   b. Set up the beaker containing acetic acid buffer/HCl solution, stir plate, and pH electrode under the buret containing the 0.2 M NaOH solution. Start gently rotating the stir bar. Record the initial buret reading and pH meter reading.

   c. Add NaOH in 1 mL increments to the buffer until the pH of the solution has increased by 3.0 pH units. Record the buret reading and the pH reading after each addition.

   d. When you have completed this titration, dispose of this solution down the sink with copious amount of water.

Clean Up

- Adjust the pH of the used solution before disposal.
  - To your 800 mL beaker, add 1 gram of sodium bicarbonate. Pour the solution down the sink with copious amount of water.
  - Consolidate the left over HCl, NaOH, acetic acid, and the buffer solutions in a 1 L bottle. Add 3 grams of sodium bicarbonate. Pour the solution in the sink with copious amount of water.
- Rinse the buret and return it to the correct storage cabinet.
- Store the pH electrode in the appropriate storage solution container.

Data Analysis

Preparing an acidic buffer

1. What is the target pH of your acidic buffer?
2. Using $M_1V_1 = M_2V_2$, calculate the volume of 2.5 M sodium acetate needed to make 250 mL of the acetic acid/acetate ion buffer that has an acetate ion concentration of 0.10 M.
3. Given the pK_a of acetic acid and a concentration of 0.10 M acetate ion, what concentration of acetic acid must be added to make a solution of your target pH?
   - Tip: Use the Henderson-Hasselbalch equation found in the introduction.
4. Using $M_1V_1 = M_2V_2$, calculate the volume of 6 M acetic acid needed to make 250 mL of the acetic acid/acetate ion buffer with the acetic acid concentration that you calculated in step 3.

Preparing a basic buffer

1. What is the target pH of your basic buffer?
2. What mass of sodium hydrogen carbonate was needed to make the buffer solution 0.050 M in sodium hydrogen carbonate? Show your calculations.
3. What was the concentration of the sodium carbonate in the hydrogen carbonate ion/ carbonate ion buffer solution? Show your calculations.
4. What mass of anhydrous sodium carbonate was required to make the 250 mL hydrogen carbonate ion/carbonate ion buffer at that pH? Show your calculations.
5. Provide reasons as to why the measured pH level is different from the calculated value.

Generating titration curves

1. Use a spreadsheet program such as Excel to enter the volumes dispensed and corresponding pH for each titration. Generate the following titration curves and make sure to label them all appropriately:
   a. pH vs. added volume of HCl for your acidic buffer solution.
   b. pH vs. added volume of HCl for your basic buffer solution.
   c. pH vs. added volume of HCl for your 0.2 M acetic acid solution.
   d. pH vs. added volume of NaOH for your acidic buffer solution.
   e. pH vs. added volume of NaOH for your basic buffer solution.
   f. pH vs. added volume of NaOH for your 0.2 M acetic acid solution.
   g. pH vs. added volume of NaOH for the acetic acid buffer/HCl mixture used in step 4 of Part IV.

   Check with your teaching assistant to see how they want the graphs turned in (i.e. by email or printed copies). Make sure your name is on each graph and that you have clearly titled and

labeled the vertical and horizontal axes. If you completed all trials, you will turn in 7 graphs total.

Comparing buffer ranges

1. Let's compare corresponding graphs. First, take the two graphs for the titrations of your acidic buffer. Within your spreadsheet program, place the graph for the titration using NaOH on the right, and the graph for the titration using HCl on the left. Select the graph on the left, and using the formatting options, flip it horizontally. Keep the pH axis (y-axis) aligned. You should now have a curve that looks like an "S" laying on its side. What is the pH range over which the buffer effectively neutralizes the added acid and base and maintains a reasonably constant pH? This is referred to as the buffer range, and is usually defined as the volume across which the buffer is able to maintain a pH equal to the pKa ± 1.

2. Repeat this procedure for the graphs involving the basic buffer. What is the buffer range for the basic buffer?

3. Repeat this procedure for the graphs involving the 0.2 M acetic acid solution. How do these graphs compare to the graphs of the acidic buffer? For example, compare the slopes of the curve of each graph at corresponding points. At corresponding pH values, compare how much HCl or NaOH is added before $\Delta pH = 1$.

To deepen your understanding of buffer systems, consider the following questions:

1. Considering the ranges of pH of each buffer, write an equation in terms pH and pK_a that defines buffer range.

2. Buffer capacity is defined as the amount of acid or base that can be added to a buffer before any substantial change in pH. When is the buffer capacity at its maximum?

3. Consider the titration curve you plotted for the "Titration of a Weak Acid" experiment. How does the titration curve compare to the graph involving the acetic acid/acetate ion buffer in this experiment? Does the titration curve include a buffer region? If so, where is the buffer region? If not, why not?

Solubility Products

Introduction

The **solubility product constant**, K_{sp}, is the equilibrium constant for a process when an ionic solid substance dissolves into an aqueous solution at a given temperature. This experiment involves the determination of the K_{sp} for calcium iodate. The calcium iodate chemical system to be analyzed is described by the reaction

$$Ca(IO_3)_2(s) \rightleftharpoons Ca^{2+}(aq) + 2IO_3^-(aq)$$

with a solubility product of

$$K_{sp} = [Ca^{2+}][IO_3^-]_2$$

In the first part of the experiment, you will determine the solubility of calcium iodate in pure water. The **solubility** (***x***) of the calcium iodate will be equal to the concentration of the calcium ion since for every mole of calcium iodate that dissolves, one mole of calcium ion forms.

Recall that the iodate ion concentration will be twice the calcium ion concentration in solution. Thus, if you can obtain the concentration of one ion you can calculate the concentration of the other ion. With the two concentrations, you can easily calculate the solubility product constant.

During Part I, you will determine the concentration of the iodate ion via what is known as an iodometric titration. In this process you will add excess iodide ion to a solution that is known to contain iodate ion in the presence of acid. The iodate reacts with the iodide by the following reaction:

$$IO_3^-(aq) + 5I^-(aq) + 6H^+(aq) \rightarrow 3I_2(aq) + 3H_2O(l)$$

The I_2 thus produced will then react via a titration with thiosulfate by the reaction:

$$I_2(aq) + 2S_2O_3^{2-}(aq) \rightarrow 2I^-(aq) + S_4O_6^{2-}(aq)$$

$$I_2(aq) + I^-(aq) \rightleftharpoons I_3^-(aq)$$

It should be noted that the progress of the latter reaction can be followed because the iodine formed reacts with the excess iodide ion to form the triiodide ion, I_3^-. The presence of this species is easily observed by its reaction with starch indicator to form a deep blue complex. Thus, in the presence of starch, the endpoint of this latter titration is when the deep blue color disappears. Once the concentration of the iodate has been determined, you can easily calculate the concentration of the calcium ion and then the K_{sp} for the system.

In the second part of this experiment, you will be able to observe the "**common ion effect**". In this part of the experiment you will be given a saturated solution of calcium iodate in a 0.01 M potassium iodate solution. Once you determine the concentration of iodate by the method described above, you will be able to calculate the concentration of iodate from the dissolution of the calcium iodate and thus calculate the concentration of calcium ion in solution.

Using the concentration of the two ions, you will be able to calculate the solubility product constant for this system. By comparing the two parts, you will note the dramatic effect that the iodate ion from the potassium iodate has on the solubility of calcium iodate. Lastly, as part of the data workup of this experiment, you will incorporate activity effects in the calculation of the solubility product from your data. The correct incorporation of activity effects makes the treatment of equilibria and equilibrium constants more rigorous.

You have discussed some of the effects of the polarity of water, including the effect that polarity can have on the solubility of solids. It should not be surprising to find that water interacts with various ions differently and that a more highly charged particle has a greater interaction with water molecules. The higher the charge on an ion in solution, the greater will be the interaction of the ion with the dipole of the water molecule and with other ions in the solution. These interactions can be significant enough that they cannot be ignored when salt concentrations exceed hundredth molar values.

Equilibrium constants are properly defined in terms of thermodynamic activity rather than concentration. The thermodynamic activity is a function of concentration, but is not necessarily equal to the concentration. However, it is true that in the limit of extremely dilute solutions, the activity is equal to the concentration. Because the equilibrium constant expressions using concentrations in place of activity are rigorously correct in the limit of dilute concentration, they are conceptually parallel with the use of activities. Because the results are useful, if not exactly correct, we commonly discuss equilibria and equilibrium constants using the concentrations. In this experiment, however, we will recognize that the true expressions are in terms of activities.

Based on the equilibrium constant of 1.3×10^{-18} for the dissolution of mercury(I) iodate, you would expect a saturated solution of the salt to be 6.9×10^{-7} M in mercury(I) ion.

$$Hg_2(IO_3)_2(s) \rightleftharpoons Hg_2^{2+}(aq) + 2IO_3^-(aq) \qquad K = 1.3 \times 10^{-18} \qquad (1)$$

$$K = [Hg_2^{2+}][IO_3^-]^2 = x(2x)^2$$

$$x = [Hg_2^{2+}] = 6.9 \times 10^{-7} \qquad (2)$$

So far, when you have determined the effect of other dissolved ions on a specific equilibrium, you have only considered the common ion effect. Based on this reasoning, you would not predict that potassium nitrate in solution would have any effect on the solubility of mercury(I) iodate. However, if you were to saturate a 0.05 M potassium nitrate with mercury(I) iodate you would find that the solubility of the mercury(I) iodate has increased by about fifty percent.

This turns out to be a general observation; any time you add an inert soluble salt to a solution of a sparingly soluble salt you will increase the solubility of the sparingly soluble salt.

The explanation for the observed increase in solubility is that the positively charged potassium ions can cluster around the negatively charged iodate ions, and the negatively charged nitrate ions cluster around the positively charged mercury(I) ions. When a mercury(I) ion comes close to an iodate ion surrounded by potassium ions, the positive charge on the potassium ions will repel the positive charge on the mercury(I) ion, preventing it from combining with the iodate ion and precipitating out of solution. Thus the mercury(I) iodate becomes more soluble.

The definition of the equilibrium constant represented in equation (2) above does not take this phenomenon into account. Instead of looking only at the concentration of a species in solution, the activity of that species should be also examined when equilibrium is considered. The activity of an ion includes both concentration and how susceptible the ion is to the kinds of effects described in the preceding paragraph. To incorporate these and other effects arising from molecular and ionic interactions in solution, we simply use the activity of the ion in place of the concentration in the equilibrium constant expression.

The general way for incorporating activities into equilibrium constants for the general reaction

$$mW + nX \rightleftharpoons pY + qZ$$

is to form the equilibrium constant in the usual way, but employing activity in positions in the equation where you were previously using concentration:

$$K = \frac{a_Y^p a_Z^q}{a_W^m a_X^n}$$

Following this procedure for our example solubility problem, equation (2) becomes:

$$K = a_{Hg_2^{2+}} + a_{IO_3^-}{}^2 \qquad (3)$$

A convenient way to quantitatively account for the molecular interaction part of the activity is to express the activity as the product of an activity coefficient times the concentration. For example, the mercury iodate equilibrium requires the activities

$$a_{Hg_2^{2+}} = \gamma_{Hg_2^{2+}}[Hg_2^{2+}]$$
$$a_{IO_3^-} = \gamma_{IO_3^-}[IO_3^-]$$

where γHg_2^{2+} and γIO_3^- are the activity coefficients for Hg_2^{2+} and IO_3^-. Substituting these expressions into equation (3):

$$K = \left(\gamma_{Hg_2^{2+}}[Hg_2^{2+}]\right)\left(\gamma_{IO_3^-}[IO_3^-]\right)^2 = \gamma_{Hg_2^{2+}} \cdot \gamma_{IO_3^-}{}^2[Hg_2^{2+}][IO_3^-]^2$$

From this form, we can see that expressing the equilibrium constant using concentrations alone is identical to assuming that the activity coefficients are equal to 1.0. This assumption is also called the **ideal solution approximation**.

Because it is impossible to get a solution containing just the cation or just the anion, it is impossible to experimentally determine γHg_2^{2+} and γIO_3^- individually. Instead their product is replaced by $\gamma_\pm$, the **mean ionic activity coefficient**, raised to the power equal to the sum of the exponents of the individual ion activity coefficients.

$$K = \gamma_\pm^3[Hg_2^{2+}][IO_3^-]^2$$

Since they account for molecular and ionic interactions, the values of activity coefficients change as the concentration of the solution changes. It has been found that a convenient quantity to use when expressing the functional dependence of the activity coefficients of ions on concentration is the ionic strength of the solution, which is defined by the expression:

$$\mu = \frac{1}{2}\sum_i C_i Z_i{}^2 \qquad (4)$$

where c_i is the concentration of the ith species and Z_i is its signed charge in multiples of the elementary charge, e.g. $Z_{Hg_2^{2+}} = +2$ and $Z_{IO_3^-} = -1$. This sum extends over all ions in solution.

In this example, when contrasting the solubility of mercury(I) iodate in pure water and in 0.05 M potassium nitrate, it becomes very clear that the ionic strength of the solution in pure water is vastly different from the solution in 0.05 M potassium nitrate when we apply this definition,

$$\text{In pure water:} \qquad \mu = \frac{1}{2}(4[Hg_2^{2+}] + [IO_3^-])$$

since Hg_2^{2+} and IO_3^- are the only ions in solution. However, in the solution containing potassium nitrate,

$$\mu = \frac{1}{2}(4[Hg_2^{2+}] + [IO_3^-] + [K^+] + [NO_3^-])$$

Because mercury(I) iodate is so sparingly soluble, calculations will give the result that in pure water, $\mu = 0.0$, whereas in 0.05 M potassium nitrate, $\mu = 0.05$.

While it is impossible to experimentally determine the values of individual ion activity coefficients, various theoretical and empirical methods for consistently separating the observed mean ionic activity coefficients into individual ion coefficients have been developed. These methods are by no means perfect, but they often give much better results than the alternative very simple assumption that the solutions are ideal. **Table 1.** presents results for one such method of representing individual ion coefficients as a function of the ionic strength of the solution.

Table 1. Activity Coefficients for Aqueous Solution at 25˚C

	Ionic Strength (μ, M)					
Ion	**0.001**	**0.005**	**0.01**	**0.05**	**0.1**	**0.15**
H^+	0.967	0.933	0.914	0.86	0.83	0.81
Li^+	0.965	0.929	0.907	0.835	0.80	0.77
Na^+, IO_3^-, HCO_3^-, $H_2PO_4^-$	0.964	0.928	0.902	0.82	0.775	0.76
OH^-, F^-, SCN^-, MnO_4^-, ClO_4^-	0.964	0.926	0.900	0.81	0.76	0.73
K^+, Cl^-, Br^-, I^-, CN^-, $NO3^-$	0.964	0.925	0.899	0.805	0.755	0.72
Rb^+, Cs^+, NH_4^+, Ag^+	0.964	0.924	0.898	0.80	0.75	0.71
Mg^{2+}, Be^{2+}	0.872	0.755	0.69	0.52	0.45	0.41
Ca^{2+}, Cu^{2+}, Zn^{2+}, Mn^{2+}	0.870	0.749	0.675	0.485	0.405	0.36
Sr^{2+}, Ba^{2+}, Cd^{2+}, Hg^{2+}, S^{2-}	0.868	0.744	0.67	0.465	0.38	0.33
Pb^{2+}, CO_3^{2-}, SO_3^{2-}	0.867	0.742	0.665	0.455	0.37	0.31
Hg_2^{2+}, SO_4^{2-}, CrO_4^{2-}, HPO_4^{2-}	0.867	0.740	0.660	0.445	0.355	0.30
Al^{3+}, Fe^{3+}, Cr^{3+}	0.738	0.54	0.445	0.245	0.18	0.15
PO_4^{3-}	0.725	0.505	0.395	0.16	0.095	0.066
Sn^{4+}	0.588	0.35	0.255	0.10	0.065	0.048

Source: Data from J. Kielland, *J. Amer. Chem. Soc.***, 59, 1675 (1937)**

As an example of how to use activities, here is a calculation of the concentration of calcium ion in a 0.0125 M solution of magnesium sulfate $MgSO_4$ saturated with calcium fluoride CaF_2. The concentration of calcium is going to depend on how much calcium fluoride dissolves, so the chemical equilibrium and initial set-up of interest is

	$CaF_{2(s)}$	$\rightleftharpoons$	$Ca^{2+}{}_{(aq)}$	+	$2F^{-}{}_{(aq)}$	$K = 3.9 \times 10^{-11}$
Initial:	*solid*		**0**		**0**	
Final:	*solid*		x		$2x$	

$$\begin{aligned} K &= (a_{Ca^{2+}})(a_{F^-}^2) \\ &= (\gamma_{Ca^{2+}}[Ca^{2+}])(\gamma_{F^-}^2[F^-]^2) \\ &= (\gamma_{Ca^{2+}}[x])(\gamma_{F^-}^2[2x]^2) \\ &= 4x^3(\gamma_{Ca^{2+}}\gamma_{F^-}^2) \end{aligned}$$

In order to look up the activity coefficients in the table, it is necessary to know the ionic strength of the solution. The ionic strength is due to the dissolved magnesium sulfate and the dissolved calcium fluoride. Since the equilibrium constant for the dissolution of calcium fluoride is quite small, assume that its contribution will be negligible, as in the earlier ionic strength calculation, and the only ions that need to be considered are the magnesium and sulfate ions. This gives an ionic strength of 0.050 M:

$$\mu = \frac{1}{2}([0.0125] \cdot 2^2 + [0.0125] \cdot (-2)^2) = 0.050M$$

Using this value for the ionic strength, the activity coefficients for calcium and fluoride ions are 0.485 and 0.81 respectively. Plugging into the equilibrium constant equation and solving for x gives

$$\begin{aligned} 3.9 \times 10^{-11} &= [x] \cdot 0.485 \cdot [2x]^2 \cdot 0.81^2 \\ x &= [Ca^{2+}] \\ &= 3.1 \times 10^{-4}\ M \end{aligned}$$

If you had neglected the activity of the ions in solution you would have calculated the calcium ion concentration to be 2.1×10^{-4} M. This is a thirty-two percent error.

In this experiment, you will examine the effect of activities in determining an equilibrium constant, the solubility product. You will do a calculation similar to the example given, but you will determine the concentrations of the species in solution, and you will use these to calculate the solubility product both with and without including activity effects.

Your solutions are not likely to have an ionic strength exactly equal to one of those given in the table. While more sophisticated interpolation between values in the table are possible, it is sufficient for this experiment to simply use the tabulated value for that ionic strength that is closest to the value you calculate for your solution of interest. If your solution has an ionic strength exactly midway between two tabulated values, then use the value for the **lower** ionic strength.

The Solvay Process

Sodium carbonate, commonly known as soda ash or washing soda, is a chemical used in many products, ranging from glass to toothpaste to moon cakes. The majority of sodium carbonate produced today comes from the Solvay process, which was developed by the Belgian chemist Ernest Solvay in the 1860s. The Solvay process takes advantage of the common ion effect to precipitate sodium bicarbonate ($NaHCO_3$) from a saturated solution of sodium chloride (NaCl), ammonia (NH_3), and carbon dioxide (CO_2). The sodium bicarbonate can then be heated to form sodium carbonate (Na_2CO_3). The Solvay process is cheaper and less environmentally harmful than older practices, which involved burning plants or producing toxic byproducts such as HCl gas. Comparatively, the Solvay process commonly uses ocean brine as a source of NaCl, limestone as a source of CO_2, and the ammonia can be recycled back into production.

Learning Goals

The following is a list of skills that you will use in this experiment.

Laboratory	• Planning and implementing a titration procedure • Using a starch indicator
Conceptual	• Solubility product constant (K_{sp}) • Solubility (x) • Activity coefficients (γ) • Ionic strength (μ) • Common Ion Effect
Data Analysis	• Calculating solubility product constants using concentration • Calculating solubility product constants using activity

Procedure

Work in pairs on this experiment.

Each student must collect data and submit a separate report. The actual data analyses and the written reports must be done entirely independently of your lab partner or other students. Make sure that you avoid unauthorized collaboration and plagiarism. All suspected violations of the Code of Academic Conduct will be referred to Student Judicial Affairs.

Stock Chemicals Used

Chemical	Maximum Amount Used
Potassium Iodate, (s)	According to calculation (< 1 g)
Potassium Iodide, (s)	According to calculation (< 7 g)
1 M Sodium Thiosulfate	According to calculation (< 15 mL)
Saturated Calcium Iodate, (aq)	< 10 mL
0.1 M Potassium Iodate in Saturated Calcium Iodate, (aq)	< 10 mL
Starch Indicator	Small amounts (< 10 mL)
6 M Hydrochloric Acid	< 6 mL

Safety First

Remember to always wear gloves when handling all solids.

Wear your goggles!

Part I. Preparation of a Potassium Iodate Solution

In this procedure you will prepare a standard potassium iodate solution for use in standardizing a sodium thiosulfate solution.

Using a volumetric flask, accurately prepare 250 mL of a solution of approximately 0.01 M KIO_3. Note that this will need to be made as accurately as possible since this solution will serve as the primary standard for the experiment. The solid will take a few moments to dissolve. While you are waiting, go on to Part II.

Safety First

The potassium iodate primary standard is being dried in the oven. Be careful! The container is extremely hot.

Always use crucible tongs to manipulate the container.

Part II. Preparation of a Sodium Thiosulfate Solution

In this procedure you will prepare and standardize a sodium thiosulfate solution. You will use the standard KIO_3 solution prepared in Part I to standardize the sodium thiosulfate solution. The sodium thiosulfate solution will then become a secondary standard.

Using your clean 1 L plastic bottle, prepare about 250 mL of a 0.05 M $Na_2S_2O_3$ solution. You should do this using the 1.0 M $Na_2S_2O_3$ stock solution provided. Some calculations are required here.

Hint

Using $M_1V_1=M_2V_2$, calculate the volume of 1.0 M sodium thiosulfate solution needed to prepare your dilute solution.

Part III. Standardization of the Sodium Thiosulfate Solution

1. Standardize the sodium thiosulfate solution with the potassium iodate solution you have prepared. You should use the iodate-to-iodine and iodine-to-iodide reactions given in the introduction of the experiment. You will have to plan how to best perform this experiment.

Green Chem

Do not waste chemicals. You should be using no more than a couple of grams of potassium iodide and a few milliliters of acid in each titration.

2. For the first reaction you will need excess potassium iodide and hydrochloric acid. If you were using a 5 mL sample of the standard potassium iodate solution it would require about 1 g of potassium iodide and about 1 mL of 6 M hydrochloric acid.

   a. Begin by dissolving the potassium iodide (KI) in about 50 mL of water in your titration flask. Next, accurately add 5.00 mL of potassium iodate using volumetric glassware. Finally, add the hydrochloric acid. The solution will turn brown due to the formation of iodine.

Tip

Do not add the starch indicator until the brown solution has lightened to a pale yellow.

   b. Immediately titrate the solution with the sodium thiosulfate solution until the solution in the titration flask becomes a pale yellow color indicating only a limited amount of iodine present. At this point add 1 mL of the starch indicator. The starch will react with the I_3^- to form a complex that is dark blue in color. Continue the titration until the dark blue color disappears. This is the end point.

3. When you have completed each titration, pour the solution in the titration flask into an 800 mL beaker.

4. Do at least three acceptable titrations so that you can calculate a meaningful average sodium thiosulfate concentration, standard deviation and 90% confidence limit for your data. An acceptable trial is one that passes the Q-test.

Titration Tips

- Do not use assembly line techniques when preparing flasks for titration; prepare a flask, titrate it, and then prepare the next flask. If you do not begin the titration immediately, iodine may crystallize out of solution and your titration results will be inaccurate.
- Do not waste time. Only your limiting reagent needs to be measured out with volumetric glassware. The other reagents in the iodate-to-iodine reaction can be measured out reasonably roughly without affecting your results.

Part IV. Solubility and Solubility Product from a Saturated Calcium Iodate Solution

1. Determine the iodate concentration in a saturated solution of calcium iodate using your standard sodium thiosulfate solution. You should use the same procedure you developed for the thiosulfate standardization, but this time you will use a saturated calcium iodate solution instead of a potassium iodate solution when performing the first reaction.

   The saturated calcium iodate solution will be provided by the stockroom. Make sure you read the labels on the bottles provided carefully; there are two calcium iodate solutions, and for this part of the experiment you are interested in the solution that has only calcium iodate dissolved in water.

2. Pour out the required amount carefully so that you do not disturb the solid $Ca(IO_3)_2$ settled at the bottom of the bottle. You do **NOT** want any of the solid $Ca(IO_3)_2$ in your Erlenmeyer flask. Pour out enough sample in a beaker so that you can conveniently but not wastefully use your 5.00 mL pipet to transfer the sample to an Erlenmeyer flask for the titration.

3. Add the same amount of KI and 6 M HCl in 50 mL of DI water that you did to standardize the thiosulfate solution, then add the 5.00 mL of saturated $Ca(IO_3)_2$ solution for the titration.

4. When you have completed this titration, pour the solution in the titration flask into an 800 mL beaker.

Part V. Solubility and K_{sp} from a Saturated Calcium Iodate Solution in 0.010 M KIO_3

1. Determine the iodate concentration in a saturated solution of calcium iodate in 0.010 M KIO_3 using standard sodium thiosulfate solution. This saturated solution is prepared by dissolving enough potassium iodate in water to make it 0.010 M in iodate and then saturating the solution with calcium iodate. This solution will also be provided by the stockroom. Again, read the label carefully; do not confuse this solution with the saturated calcium iodate solution you used in the previous part of this experiment.

2. Pour out the required amount carefully so that you do not disturb the solid $Ca(IO_3)_2$ settled at the bottom of the bottle. You do NOT want any of the solid $Ca(IO_3)_2$ in your Erlenmeyer flask. Pour out enough sample in a beaker so that you can conveniently but not wastefully use your 5.00 mL pipet to transfer the sample to an Erlenmeyer flask for the titration. Add the same amount of KI and 6 M HCl in 50 mL of DI water that you did to standardize the thiosulfate solution, then add

the 5.00 mL of saturated $Ca(IO_3)_2$ in 0.010 M KIO_3 solution for the titration.

3. When you have completed this titration, pour the solution in the titration flask into an 800 mL beaker.

Clean Up

- Pour any remaining HCl into the 800 mL beaker. Slowly and carefully add 3 grams of sodium bicarbonate. Pour this solution down the sink with copious amounts of water.

Data Analysis

Standardization of $Na_2S_2O_3$

1. The following equations represent two reactions that take place during the iodometric titration and standardization of your sodium thiosulfate. The iodate from KIO_3 reacts with iodide and acid to form iodine. The iodine then reacts with the thiosulfate from $Na_2S_2O_3$. Fill in the stoichiometric values to determine the number of moles of $Na_2S_2O_3$ that react in the presence of one mole of KIO_3. **Make sure to balance the charge!**

$$\_\_\ IO_3^-(aq) + \_\_\ I^-(aq) + \_\_\ H^+(aq) \rightarrow \_\_\ I_2(aq) + \_\_\ H_2O(l)$$

$$\_\_\ I_2(aq) + \_\_\ S_2O_3^{2-}(aq) \rightarrow \_\_\ I^-(aq) + \_\_\ S_4O_6^{2-}(aq)$$

2. For each of your three trials, use the volume and molarity of KIO_3 to determine the number of moles of KIO_3 in solution.

3. For each of your three trials, use the number of moles of KIO_3 present in solution and the stoichiometric ratio between KIO_3 and $Na_2S_2O_3$ to determine the number of moles of $Na_2S_2O_3$ in solution.

4. For each of your three trials, use the volume of $Na_2S_2O_3$ at the equivalence point and the number of moles of $Na_2S_2O_3$ present in solution to determine the molarity of your $Na_2S_2O_3$ solution.

5. What is the average molarity, standard deviation, and 90% confidence limit of the $Na_2S_2O_3$ solution based on your three trials?

Calculating solubility (x) of $Ca(IO_3)_2$ in pure water

1. In your titration of saturated $Ca(IO_3)_2$ solution in pure water, what volume of standardized Na_2SO_3 was required to reach the endpoint?

2. Use the volume and molarity of your standardized $Na_2S_2O_3$ to determine the number of moles of $Na_2S_2O_3$ dispensed at the equivalence point.

3. Use the number of moles of $Na_2S_2O_3$ at the equivalence point and the stoichiometric ratio between $Na_2S_2O_3$ and KIO_3 to determine how many moles of IO_3^- were present in solution.

4. Use the number of moles of IO_3^- and the volume of saturated $Ca(IO_3)_2$ in water to calculate the molarity of IO_3^-.

5. Based on the molarity of IO_3^- present in your sample, what is the solubility (x) of $Ca(IO_3)_2$ in pure water?

    - Tip: the solubility (x) of calcium iodate will be equal to the concentration of the calcium ion since for every mole of $Ca(IO_3)_2$ that dissolves, one mole of Ca^{2+} forms. It may help to set up an ICE table detailing the dissociation of $Ca(IO_3)_2$ in water to see how the concentration of IO_3^- and Ca^{2+} are related.

Calculating solubility product (K_{sp}) of $Ca(IO_3)_2$ in pure water based on concentration

You will start your analysis of solubility product constants by calculating K_{sp} of $Ca(IO_3)_2$ using only concentration. As you read in the Introduction, this method of calculating K_{sp} assumes that the activity coefficients (γ) of each ion is equal to 1.

1. Write an equation that describes the solubility product, K_{sp}, of $Ca(IO_3)_2$ in pure water in terms of the concentration of Ca^{2+} and IO_3^-.
2. Re-write this equation by substituting solubility (x) of $Ca(IO_3)_2$ in for the concentrations of Ca^{2+} and IO_3^-. Your equation should now be written in terms of x.
3. Use the solubility (x) of $Ca(IO_3)_2$ that you determined in step 5 of "Calculating solubility (x) of $Ca(IO_3)_2$ in pure water" to solve for the solubility product, K_{sp}, of $Ca(IO_3)_2$.

Calculating solubility product (K_{sp}) of $Ca(IO_3)_2$ in pure water based on activity

Now you will do a more in-depth calculation of solubility product constants by calculating K_{sp} of $Ca(IO_3)_2$ using both concentration and activity. This method of calculating K_{sp} takes into account interactions between ions in solution.

1. Use equation (4) from the Introduction to write out an equation calculating the ionic strength (μ) of $Ca(IO_3)_2$ in pure water using the concentration (c_i) and charge (Z_i) of each ion.
2. Re-write this equation by substituting solubility (x) of $Ca(IO_3)_2$ in for the concentrations of Ca^{2+} and IO_3^-. Your equation should now be written in terms of x.
3. Use the solubility (x) of $Ca(IO_3)_2$ you previously calculated to solve for the ionic strength ($\boldsymbol{\mu}$) of $Ca(IO_3)_2$ in pure water.
4. Use Table 1 in the Introduction and your calculated ionic strength ($\boldsymbol{\mu}$) to find the activity coefficients (γ) for Ca^{2+} and $Ca(IO_3)_2$.
5. Write an equation that describes the solubility product, K_{sp}, of $Ca(IO_3)_2$ in pure water in terms of both the concentration and activity coefficients (γ) of Ca^{2+} and IO_3^-.
6. Re-write this equation by substituting solubility (x) of $Ca(IO_3)_2$ in for the concentrations of Ca^{2+} and IO_3^-. Your equation should now be written in terms of x and γ.
7. Use the solubility (x) of $Ca(IO_3)_2$ and activity coefficients (γ) of Ca^{2+} and IO_3^- that you determined previously to solve for the solubility product, K_{sp}, of $Ca(IO_3)_2$ in pure water.

Determining solubility (x) of $Ca(IO_3)_2$ in 0.01 M KIO_3

You will now study the Common Ion Effect by calculating the solubility of $Ca(IO_3)_2$ in a solution already containing 0.01 M KIO_3. When you do this calculation, make sure that you account for the fact that 0.010 M of IO_3^- does not come from the dissolved $Ca(IO_3)_2$, but the KIO_3 instead.

1. In your titration of saturated $Ca(IO_3)_2$ solution in 0.01 M KIO_3, what volume of standardized Na_2SO_3 was required to reach the endpoint?

2. Use the volume and molarity of your standardized $Na_2S_2O_3$ to determine the number of moles of $Na_2S_2O_3$ dispensed at the equivalence point.

3. Use the number of moles of $Na_2S_2O_3$ at the equivalence point and the stoichiometric ratio between $Na_2S_2O_3$ and KIO_3 to determine how many moles of IO_3^- were present in solution.

   - Tip: the moles of IO_3^- present in this solution are from both the dissociation of $Ca(IO_3)_2$ and the dissociation of 0.01 M KIO_3.

4. Use the number of moles of IO_3^- and the volume of saturated $Ca(IO_3)_2$ in 0.01 M KIO_3 to calculate the molarity of IO_3^-.

5. Based on the molarity of IO_3^- present in your sample, what is the solubility (x) of $Ca(IO_3)_2$ in 0.01 M KIO_3?

   - Tip: you must take into account the fact that 0.01 M of the total IO_3^- concentration comes from the dissociation of 0.01 M KIO_3. This means that the total concentration of IO_3^- you calculated above is equal to 0.01M + 2x.

Calculating solubility product (K_{sp}) of $Ca(IO_3)_2$ in 0.01 M KIO_3 based on concentration

1. Write an equation that describes the solubility product, K_{sp}, of $Ca(IO_3)_2$ in pure water in terms of the concentration of Ca^{2+} and IO_3^-.

2. Re-write this equation by substituting solubility (x) of $Ca(IO_3)_2$ in for the concentrations of Ca^{2+} and IO_3^-. Your equation should now be written in terms of x.

   - Tip: you must take into account the fact that 0.01 M of the total IO_3^- concentration comes from the dissociation of 0.01 M KIO_3. This means that the total concentration of IO_3^- is equal to 0.01M + 2x.

3. Use the solubility (x) of $Ca(IO_3)_2$ that you determined in step 5 of "Calculating solubility (x) of $Ca(IO_3)_2$ in 0.01 M KIO_3" to solve for the solubility product, K_{sp}, of $Ca(IO_3)_2$.

Calculating solubility product (K_{sp}) of $Ca(IO_3)_2$ in 0.01 M KIO_3 based on activity

1. Use equation (4) from the Introduction to write out an equation calculating the ionic strength (μ) of $Ca(IO_3)_2$ in 0.01 M KIO_3 using the concentration (c_i) and charge (Z_i) of each ion, **including K^+**.

2. Re-write this equation by substituting solubility (x) of $Ca(IO_3)_2$ in for the concentrations of Ca^{2+}, IO_3^-, and 0.01 M for the concentration of K^+. Your equation should now be written in terms of x.

   - Tip: you must take into account the fact that 0.01 M of the total IO_3^- concentration comes from the dissociation of 0.01 M KIO_3. This means that the total concentration of IO_3^- is equal to 0.01M + 2x.

3. Use the solubility (x) of $Ca(IO_3)_2$ you calculated in step 5 of "Calculating solubility (x) of $Ca(IO_3)_2$ in 0.01 M KIO_3" to solve for the ionic strength (**μ**) of $Ca(IO_3)_2$ in 0.01 M KIO_3.

4. Use Table 1 in the Introduction and your calculated ionic strength (**μ**) to find the activity coefficients (γ) for Ca^{2+} and IO_3^-.

5. Write an equation that describes the solubility product, K_{sp}, of $Ca(IO_3)_2$ in 0.01 M KIO_3 in terms of both the concentration and activity coefficients (γ) of Ca^{2+} and IO_3^-.

6. Re-write this equation by substituting solubility (*x*) of $Ca(IO_3)_2$ in for the concentrations of Ca^{2+} and IO_3^-. Your equation should now be written in terms of *x* and γ.

   - Tip: you must take into account the fact that 0.01 M of the total IO_3^- concentration comes from the dissociation of 0.01 M KIO_3. This means that the total concentration of IO_3^- is equal to 0.01M + 2*x*.

7. Use the solubility (*x*) of $Ca(IO_3)_2$ and activity coefficients (γ) of Ca^{2+} and IO_3^- that you determined previously to solve for the solubility product, K_{sp}, of $Ca(IO_3)_2$ in 0.01 M KIO_3.

Chem 2 Series Laboratory

Procedures and Safety Handbook

Revision Date: August 2023

Appendix Table of Contents

General Experimental Guidelines

The laboratory is a critical component of your study of chemistry. Therefore, a student must complete **all of the assigned laboratory work**, including all on- & off-line post-laboratory exercises, in order to pass this course.

1. Pre-Laboratory Preparation

- You are required to prepare for each experiment by doing the following:
- Carefully read the experiment and write a Title, Purpose, Procedure (brief outline), and Data (outline) section before arriving at the laboratory. A detailed description of each section is described below under, "Writing a Laboratory Report".
- You must complete the on-line pre-laboratory presentation and must pass the pre-laboratory quiz.

If you have not completed the pre-lab preparation at the beginning of the laboratory period, you will be deemed unsafe to perform the experiment and must leave the laboratory until the pre-laboratory write up is complete and your TA is convinced that you are prepared to begin the experiment.

2. Data Collection

All data must be recorded *in blue or black ink* directly into your laboratory notebook. At the completion of the experiment, you must turn in a copy of your data sheet to your TA *before* you leave the laboratory.

3. Unknowns

Students will obtain all unknowns from the TA. Students must be explicit in their request for an unknown; that is, they must know the name of the experiment and unknown. If a student needs more unknown, they should notify the TA who will then write a note of explanation that the student can take to the dispensary. The note should contain the student's name, the student's locker number, the laboratory section number, the TA's name, the experiment name, and the name of the unknown.

4. Writing A Laboratory Report

Below is the suggested format that your report should follow. Portions of the report should be written in your laboratory notebook and others will be submitted on-line as part of the post laboratory exercises. Post laboratory exercises are due one week after the completion of the laboratory.

Below is a general outline of a common format that is often used in science laboratory courses. Discuss this format with your TA during the first laboratory period so that you clearly understand what will be expected. All reports must be written in **non-erasable blue or black ink**. A date should be indicated on each report. Your notebook should be organized and written in such a manner that another chemist could read it and repeat the experiment in precisely the same way.

- **Title:** The report should have a title that concisely describes the experiment.
- **Purpose:** A brief and concise statement that describes the goals of the experiment and the methods employed. Any pertinent chemical reactions are generally indicated. State the purpose of the experiment in the form of a complete sentence. Do not start with the word "To."
- **Procedure:** A brief and concise outline of each step of the experiment should be included. If you are using a published procedure, you should also cite the literature or laboratory manual. A drawing of the apparatus may also be included.
- **Data and Observations:** Report all measurements and observations that 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 in **blue or black ink** directly into the notebook at the time it is collected. A severe penalty will be imposed for pencil or transcribed data entries. Do not erase mistakes. Simply draw a line through the error and record the correction. Your notebook is subject to examination at any time.

The following sections are to be submitted on-line as part of the post-laboratory exercise. You should complete the post-lab report as soon as possible after the completion of the experiment as this is much more efficient than waiting until the night before the experiment is due.

- **Calculations:** This section generally includes any complicated calculations that are involved in the experiment. Again, it is important to use foresight when organizing this section.
- **Questions:** All assigned questions are answered in this section.
- **Results & Conclusions:** Report the outcome of the experiment.

Laboratory Work Grading Policies

1. **Pre-lab lab notebook preparation incomplete:**
   - 30% of post-lab score deduction for first offense.
   - 70% of post lab score deduction for subsequent offenses.
   - No extra time or make-up
2. **Online Pre-lab quiz failed or incomplete 1 hour before lab begins:**
   - 0/2 points for the pre-lab quiz
3. **Late reports**
   - 5-point deduction for every calendar day the report is late

Late Reports & Make-Up Policy

1. Late Reports

Laboratory reports are due at the beginning of the period after the one allocated for the completion of the experiment. The last report each quarter is due at the time indicated by the TA. Late reports will be met with a 5-point deduction for every calendar day the report is late.

2. Laboratory Make-Up Policy

You must attend the laboratory class for the section in which you are enrolled. **If you miss a laboratory class with an excused absence, it must be made up before the end of the following week of laboratory. No laboratory make-ups will be offered after one week from the scheduled date of the lab.** If you miss the last lab of the quarter, it must be made up **immediately**. No make ups for unexcused absences.

Excused absences include an extended illness, accidents, or family emergencies. Vacation, cruises, and IM sports are not considered excused. Bring documented proof of your excused absence to your TA or head TA immediately upon return. If you cannot present this documentation or have an unexcused absence, you may receive a failing grade in the course.

You are required to complete all labs in order to pass the course and it is your responsibility to find an open laboratory in the same course promptly. Failure to make up a lab may result in a **failing grade** for the course.

3. Laboratory Make-up Procedure

If you miss a lab, you must make it up by attending another scheduled laboratory section. It is your responsibility to find an open laboratory in the same course. Consult the Class Schedule and Room Directory for a listing of rooms and times. Go to the selected laboratory section and ask the teaching assistant if you may be admitted to make up a lab. You must be on time for the start of the lab period. If there is room in the class, the teaching assistant will allow you in the lab, unlock your locker, and allow you to do the lab. Make sure to record the **teaching assistant's name, date, time and room number** where you made up the laboratory. Have the TA collect your data sheet and he or she will give it to your regularly assigned teaching assistant. **No laboratory report will be accepted without a valid copy of the data sheet.**

4. Plagiarism and Unauthorized Collaboration

Some of your experiments will be done with lab partners. You are encouraged to discuss your data and its analysis and interpretation with your lab partner, other students and the TAs. However, the actual data analyses and the written reports **must** be done entirely independently of your lab partner or other students. Make sure that you avoid unauthorized collaboration and plagiarism. All suspected violations of the Code of Academic Conduct will be referred to Student Judicial Affairs.

Chemistry Department Safety Policy

U.C. Davis Department of Chemistry Chem. 2 Series
Standard Operating Procedures
SAFETY RULES FOR TEACHING LABORATORIES

The following rules are designed for your safety in the laboratory. The Laboratory Instructor (LI = TA, Laboratory Supervisor, and/or Course Instructor) is required to enforce these rules and has the full backing of the Department of Chemistry Staff and Faculty. The LI is also required to enforce all laboratory experiment-specific safety procedures in carrying out the laboratory work. Violations of these rules will result in expulsion from the laboratory.

1. **No one is allowed in the laboratory without the supervision of a LI. No laboratory work will be done without supervision. Perform only authorized experiments, and only in the manner instructed. DO NOT alter experimental procedures, except as instructed.**
2. **Specific permission from your LI is required before you may work in any laboratory section other than the one to which you have been assigned.** Only laboratory rooms where the same laboratory course is operating may be used for this purpose.
3. **If you have a special health condition (asthma, pregnancy, etc.) or any personal health concerns, consult your medical professional before taking chemistry lab.**
4. **If you come to the laboratory with non-compliant goggles, shoes, or clothing, you will not be allowed to work in the laboratory. In that context, note** THERE ARE NO MAKE-UP LABORATORIES**.** Your course grade will be significantly lowered or you may fail the course if you do not meet the lab attire requirements.
5. **100% cotton lab coats are REQUIRED.**
6. **Approved safety goggles must be worn by all persons at all times.** At NO TIME are safety glasses of any kind acceptable in the laboratory. Safety goggles may not be modified in any manner.
7. **Clothing that completely covers the legs—including the skin between the top of the shoe and the bottom of the pant leg—must be worn at all times in the laboratory** (tights or leggings are NOT suitable leg covering). Inadequate protection often leads to injury. Avoid wearing expensive clothing to lab as it may get damaged.
8. **Closed-toe, closed-heel shoes that completely cover the entire foot must be worn at all times.**
9. **Confine long hair while in the laboratory.**
10. **Horseplay and carelessness are not permitted and are cause for expulsion from the laboratory. You are responsible for everyone's safety.**
11. **Absolutely NO food or drinks are to be stored or consumed in the laboratory.** Contact lenses and cosmetics (hand lotion, lip balm, etc.) are not to be applied and medications are not to be consumed while in the laboratory.

12. Skateboards, rollerblades, and other such personal equipment must be stored outside of the laboratory. Personal electronics are only permitted when needed for the laboratory. Because cell phones or other personal electronic media can easily be damaged or contaminated in the laboratory, use of such devices is at the student's own risk.
13. **Learn the location and how to operate the nearest eyewash fountain, safety shower, fire extinguisher, and fire alarm box.** Basic first aid for any chemical splash is to wash the affected area for at least 15 minutes and seek medical attention. Use the emergency shower if appropriate, removing contaminated clothing for thorough washing. If the safety shower or eyewash is activated, the exposed person should be accompanied to the Student Health Center for further evaluation.
14. **Laboratory doors must remain closed except when individuals are actively entering or exiting the lab.**
15. **The student must have at least ONE UNGLOVED HAND when outside the laboratory.** Gloves are presumed to be contaminated and must not come into contact with anything outside the laboratory except chemical containers. Only use the **ungloved hand** to open doors, hold on to stair rails, or push elevator buttons.
16. **All activities in which toxic gases or vapors are used or produced must be carried out in the fume hood.**
17. **Mouth suction must never be used to fill pipets.**
18. **Containers of chemicals may not be taken out of the laboratory except to the dispensary for refill/replacement or to exchange full waste jugs for empty ones.** All containers must be **closed with the appropriate cap** before you take them into the hallway to the dispensary. Always use a bottle carrier when transporting chemicals and waste.
19. **Put all hazardous waste into the appropriate waste container(s) provided in your laboratory.** Do not overfill waste containers.
20. **All incidents, near misses, injuries, explosions, or fires must be reported at once to the LI.** In case of serious injury or fire (even if the fire is out), the LI or Lab Supervisor must call 911. The student must always be accompanied to the Student Health Center.
21. **Keep your working area clean – immediately clean up ALL spills or broken glassware. Dispose sharps in the appropriate container. Do not dispose pipette tips in regular trash.** Clean off your lab workbench before leaving the laboratory.

You must sign the Safety Acknowledgement sheet before you may work in the lab. If you have questions about these rules and procedures, please ask your LI before starting any laboratory work in this course.

Safety in the Chemistry 2 Laboratories

Students are an integral part of accident and injury prevention effort. The laboratory safety rules require the students to follow Safe Laboratory Practices and wear the proper **Personal Protective Equipment (PPE)**.

Safe Laboratory Practices

Using safe laboratory practices prevents most accidents and injuries from occurring. Remember that you are sharing the same work area with 23 other students. Any unsafe practices on the part of your fellow students may end up injuring you or others. Courteously correct unsafe lab practices you may encounter or report them to your TA. Laboratory safety is a communal effort.

1. Work Under Supervision

Your TA must be present to supervise all experiments. If your TA is incapacitated, contact dispensary staff immediately.

Report all accidents and injuries to your TA, no matter how small.

2. Follow Instructions

The Chemistry 2 laboratory is designed to minimize the hazard exposure to students. Failure to follow the lab manual instructions may result in accidents and injuries to you and others around you.

Always follow the manual unless directly instructed by your Laboratory Instructor or the teaching lab staff.

Follow all instructions posted in the laboratory.

3. Safety Equipment

There are many safety types of equipment in the Chemistry 2 laboratory. Learn where they are and how to operate them.

- **Exits**

  The ability to remove yourself from a dangerous situation is one of the most important safety skills you have.

  Keep the exits clear. Do not block exits with backpacks, skateboards, bicycles, etc.

  Keep the doors closed. Do not prop the door open.

- **Fire Extinguisher**

  Learn the location of the fire extinguisher. It is usually placed next to an exit.

- **Eyewash**

  Learn the location of the eyewash. For chemical spills in your eyes, use the Eyewash fountain. Hold your eyelids open and wash affected area water for 15 minutes with water. Seek medical attention.

- **Drenching Hose and Safety Shower**

  Learn the location of the drench hose and safety shower.

  For large spills on your body, use the **safety shower**.

  - Remove contaminated clothing and wash affected area with water. Seek medical attention immediately.
  - When the safety shower is used, all other students must evacuate the room.

  The TA **must** dial 911 and inform the Fire Department that the safety shower is used.

  For small chemical spills on your arms and hands, use the **drench hose**.

  - Wash affected area water for 15 minutes with water and contact your TA. You may also use the tap water faucet if it is adequate for washing the affected area. It is advised that you seek medical attention for even minor burns.

- **Fire Alarm Box**

  The fire alarm boxes in the Science Lab building are located in the hallway.

4. Practice Good Housekeeping

Keep work area organized. Don't put glassware on edges where they may fall off.

Cap all bottles and close all drawers immediately.

Clean up all spills and broken glassware immediately.

5. Avoid Chemical Contamination

Do not bring food and drinks into labs.

Do not consume or use food, beverage, medicine, chewing gum, or tobacco, apply makeup or contact lenses in the laboratory.

Take off one glove when leaving the laboratory. Do not touch anything outside the laboratory with your laboratory gloves.

Wash your hands thoroughly before leaving the lab.

Personal Protective Equipment (PPE)

Students must come to the laboratory section with the appropriate personal protective equipment. The PPE is the last line of defense against chemical hazards in the laboratory. Failure to don the appropriate PPE will result in your removal from the laboratory. Many students may find it helpful to keep a bag dedicated to chemistry lab courses with the proper clothing and PPE and change into them before class.

1. Dress Code

Clothing worn in the laboratory should be able to protect you from small splashes and spills of liquids. For the Chemistry 2 laboratories, students are required to have long sleeves, long pants, and shoes that cover the entirety of the foot.

- **Long sleeve shirt and long pants:**

  You must wear clothing that covers your arms, legs, wrists and ankles to protect you from small spills. Long skirts, tights or leggings do not qualify. Do not wear clothing with holes in them as they will not protect you from spills.

- **Shoes that cover the entirety of the foot and socks to cover the ankles:**

  You must wear closed-toe, closed-heeled shoes that completely cover your foot. Do not wear sandals, slippers, or shoes that expose the back of your foot. Broken glassware and spilled chemicals are more likely to land on your foot than anywhere else. Also remember to wear socks to cover your ankles. The area between your shoes and pants should not be exposed when you are seated.

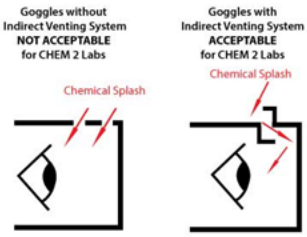
  A good rule of thumb to keep in mind is: **No skin exposure from the neck down to the feet in the laboratory.**

2. Goggles

Lab goggles are designed to protect your eyes. Injury to the eyes is often irreversible and may severely impact your future. Always wear approved goggles when working in the laboratory.

- **Approved Goggles**

  ANSI Z87.1-compliant chemical splash goggles with indirect venting is required for the Chemistry 2 course. Approved lab goggles may be purchased at the MU Bookstore, the Silo Bookstore or the ARC Pro Shop in the Activity and Recreation Center.



- **Goggles Rules**

  Modified goggles will not be allowed in the lab. Do not modify the goggles by adjusting or removing the indirect venting system.

  Goggles strap must be adjusted to fit properly at all times.

  Never take off your goggles in the laboratory. If you need to adjust your goggles or if they fog up, leave the laboratory and return when your goggles issues are resolved.

3. Lab Coat

You must provide your own lab coat for all chemistry lab courses. Only wear lab coats during the laboratory. Take off your lab coat immediately after lab. Do not wear lab coat outside the laboratory.

Your lab coat must be made of 100% cotton. Disposable, synthetic lab coats are not acceptable.

Your lab coat must be properly fitted so that it protects your arms and body. The sleeves of your lab coat must fully extent to the wrists. Do not wear a lab coat that that's too small or too big for you.

Keep your lab coat buttoned at all times.

4. Gloves

You will be provided with disposable nitrile gloves in lab for you protection. Do not bring your own gloves.

Wear gloves when handling hazardous chemicals or contacting potentially contaminated surfaces.

Never re-use disposable gloves. **Remove and replace contaminated nitrile gloves immediately.**

- **Allergy**

  If you are allergic to nitrile gloves, contact your TA and the laboratory staff. You will be provided with hypoallergenic lab gloves.

- **Fit**

  Make sure you wear the correct sized gloves. Gloves that are too large for your hand greatly increase the likelihood of accidents.

Maps and Emergency Evacuation Procedures

1. Prior to Exiting

After being notified to evacuate, cease all work activities and evacuate immediately.

Stop all reactions and turn off all sources of ignition.

Close, but do not lock, the doors. Take your purse, briefcase, backpack and keys with you if possible. Remember that you may not be allowed back into the building for an extended time.

2. Evacuation Routes/Exiting the Building

During an emergency evacuation, use the nearest door or stairway if available to exit the building. Do not use elevators for fire/earthquake evacuations.

Be aware of at least two exit routes in the event one is compromised.

3. Assembly Area

After exiting the building, all occupants should follow the evacuation route to the pre-arranged assembly area.

DO NOT return to the building until notified by emergency personnel. Supervisors must take roll to ensure all occupants have safely evacuated the building.

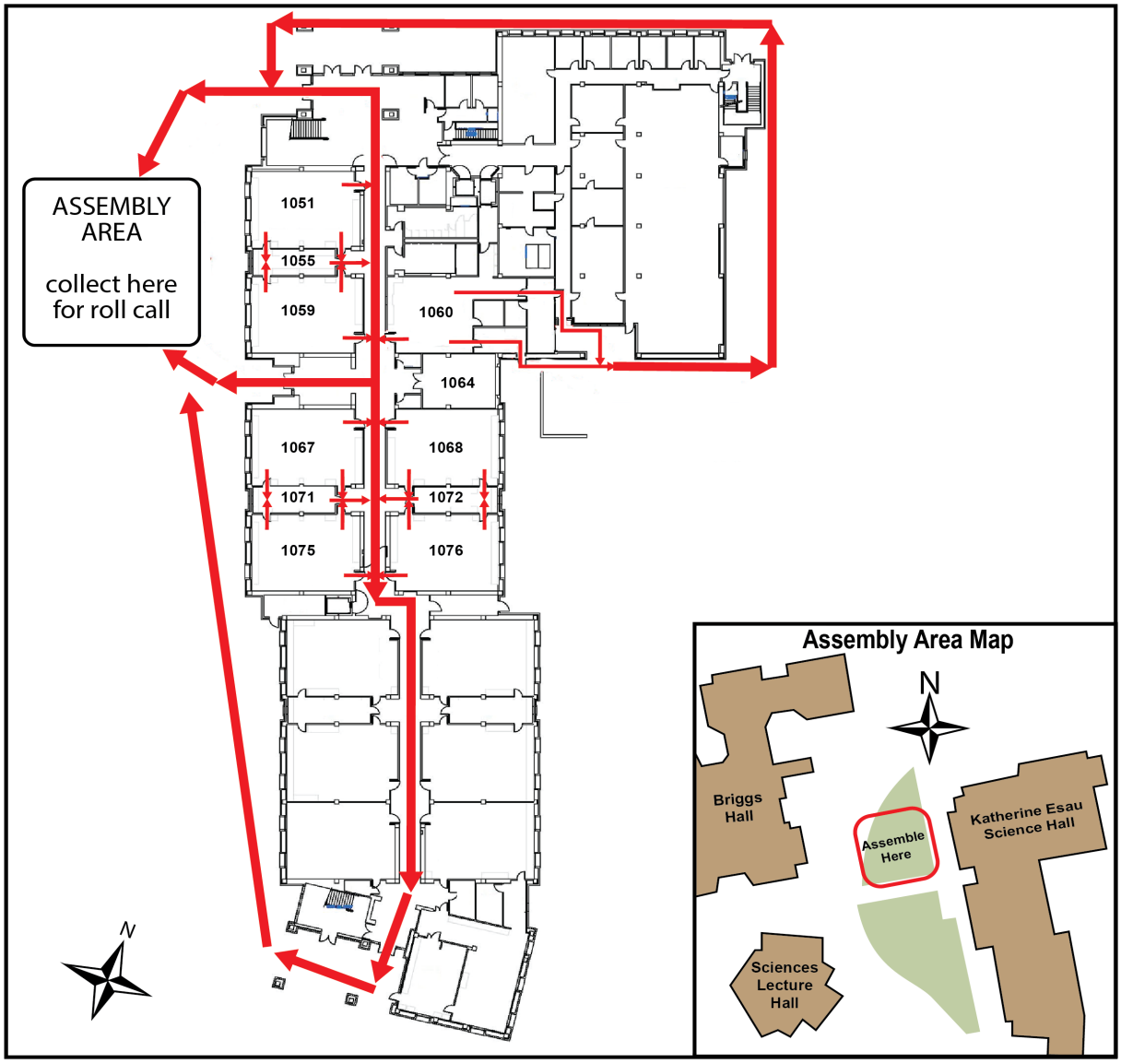


Figure 1. Evacuation routes for the 1st floor Esau Hall rooms.

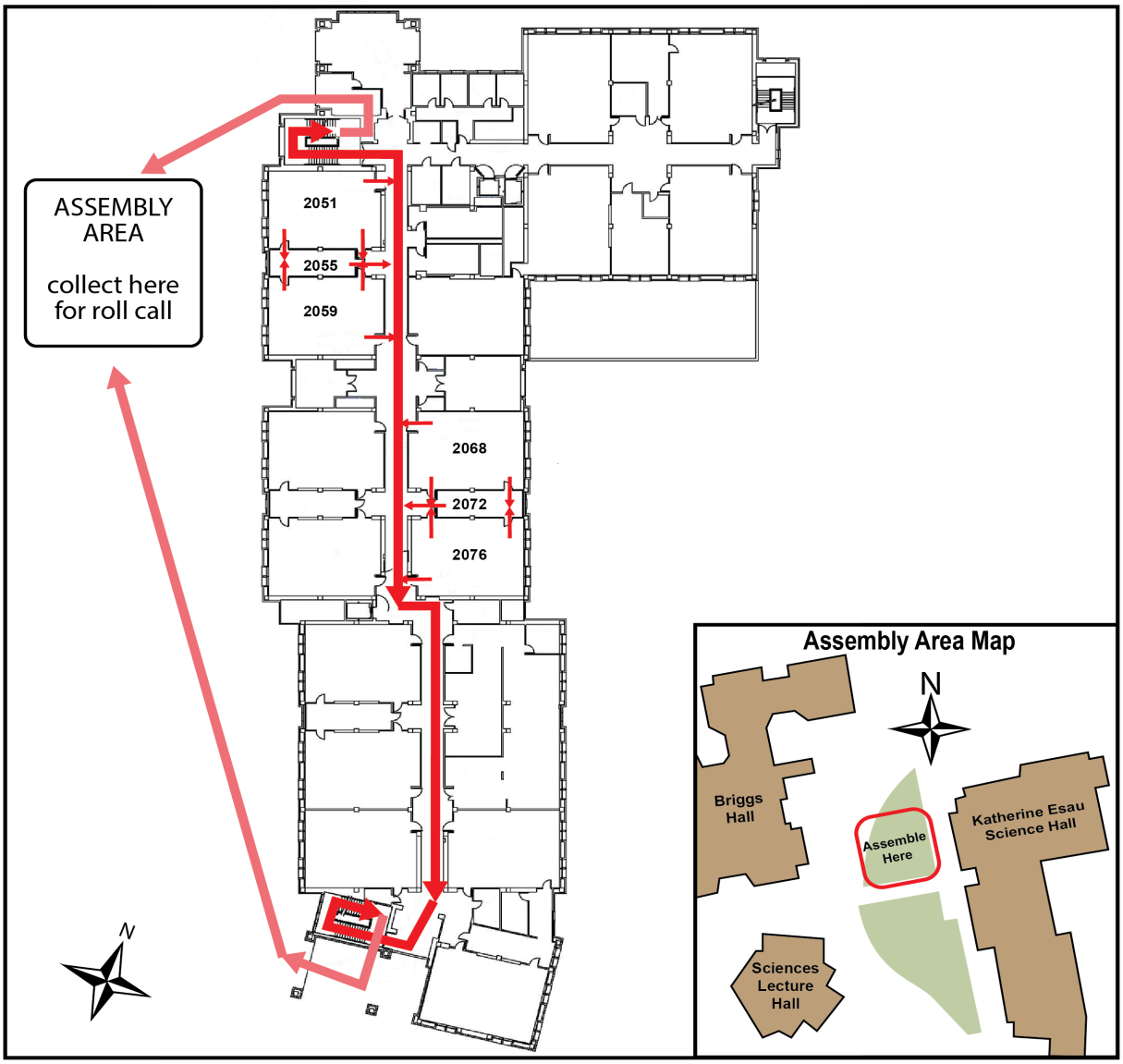


Figure 2. Evacuation routes for the 2nd floor Esau Hall rooms.

Figure 3. The assembly area for Chemistry 2 students and personnel.

General Emergency Procedures

The following are some general instructions for actions to take in case of an emergency:

1. Medical Emergency

1) Remain calm.
2) Initiate lifesaving measures if required.
3) TA will call for the dispensary supervisor and/or for Emergency Response—CALL 911.
4) Do not move injured persons unless it is necessary to prevent further harm.
5) Keep injured person warm.

2. Major Incident

1) Alert TA to injured or contaminates persons.
2) Alert people to evacuate the area.
3) TA will call for the dispensary supervisor and/or Emergency Response—CALL 911.

   Fire.....911

   Chemical, radiation, biological spill.....911

   (Evenings and Weekends).....911

4) Close doors to affected areas.
5) Have person knowledgeable of incident inform the TA.

3. Fire Alarm

1) When fire alarm sounds, evacuate the room and follow evacuation plan to the Assembly Area. The Assembly Area is on the south side of the large tree, which is on the west side of the Esau Hall.
2) TAs must take roll to ensure all students are accounted for.
3) If the building is cleared, you will return to continue your lab.

Dispensary Procedures

1. Dispensary Location and Policies

The CHE2 dispensary is located on the first floor of the Esau Hall in **Room 1060**. Go to the **dispensary roll-up window (1060E)** for service.

You must wear the proper PPE to the dispensary. This includes your **lab coat and goggles**. Remember that you should have at least **one ungloved hand** while outside your laboratory.

2. Dispensing Policies

a.) Policies at the Beginning of the Quarter

Goggles and Lab Coat: You are required to provide your own goggles and lab coats.

Locker Supplies: It is required that you do a locker inventory during the first week of labs. Make sure that you have everything on your locker list by the end of the second week of instruction.

b.) Policies During the Quarter

Locker Supplies: If a locker item is broken after the initial two-week period, go to the dispensary to request a replacement. You must know the exact name and specification of the item to be replaced.

Refilling of Chemical and Supply Containers: When replacing or refilling general laboratory chemicals or supplies, be sure to bring the empty containers to the dispensary. Be sure all containers are closed with the correct cap and placed in the correct bottle carrier.

To avoid chemical contamination and equipment breakage, please refrain from bringing personal bags and backpacks to dispensary window when seeking replacement chemical containers or lab equipment.

Waste Containers: Call the dispensary for replacements when waste containers are full.

c.) Policies at the End of the Quarter

Surplus Stores: Any item you may have in surplus should be placed in the area set aside for surplus items in the laboratory (a box at the back of the lab).

Filling Locker Requirements: If your locker is short of any items when you are checking your locker equipment against your locker list, obtain the missing items from the surplus items in the laboratory. If the missing item is not in the surplus area, obtain it from the dispensary.

Preparing Your Locker for Check-Out: Clean and quickly dry all equipment. Replace all broken or missing items by checking them out from the dispensary. Return all extra equipment to the extra glassware box in the lab. Have your TA check the contents of the locker and if everything is present and clean then they will lock the drawer.

Safety Data Sheet

The Safety Data Sheet (SDS) is a document that provides information to enable users of a substance or mixture to take the necessary measures relating to protection of health and safety at the workplace, and the protection of the environment. A Safety Data Sheet has the following sections:

1. Identification:
2. Hazard identification;
3. Composition/information on ingredients;
4. First-aid measures;
5. Fire-fighting measures;
6. Accidental release measures;
7. Handling and storage;
8. Exposure controls/personal protection;
9. Physical and chemical properties;
10. Stability and reactivity;
11. Toxicological information;
12. Ecological information;
13. Disposal considerations;
14. Transport information;
15. Regulatory information;
16. Other information.

A list of SDS resources may be found at: http://ehs.ucop.edu/sds

The following pages show a sample SDS for the **6 M Hydrochloric Acid** commonly used in the CHE2 laboratory courses.

Fisher Scientific

Part of Thermo Fisher Scientific

SAFETY DATA SHEET

Creation Date 24-Aug-2009 **Revision Date** 24-Feb-2014 **Revision Number** 1

1. Identification

Product Name **Hydrochloric Acid Solution, 6N (Certified)**

Cat No. : **SA56-1; SA56-4; SA56-200; SA56-500**

Synonyms Chlorohydric acid; Hydrogen chloride solution.; Muriatic acid

Recommended Use Laboratory chemicals.

Uses advised against No Information available

Details of the supplier of the safety data sheet

Company
Fisher Scientific
One Reagent Lane
Fair Lawn, NJ 07410
Tel: (201) 796-7100

Emergency Telephone Number
CHEMTREC®, Inside the USA: 800-424-9300
CHEMTREC®, Outside the USA: 001-703-527-3887

2. Hazard(s) identification

Classification

This chemical is considered hazardous by the 2012 OSHA Hazard Communication Standard (29 CFR 1910.1200)

Corrosive to metals	Category 1
Skin Corrosion/irritation	Category 1 B
Serious Eye Damage/Eye Irritation	Category 1
Specific target organ toxicity (single exposure)	Category 3
Target Organs - Respiratory system.	

Label Elements

Signal Word
Danger

Hazard Statements
May be corrosive to metals
Causes severe skin burns and eye damage
May cause respiratory irritation

Precautionary Statements
Prevention
Do not breathe dust/fume/gas/mist/vapors/spray
Wash face, hands and any exposed skin thoroughly after handling
Wear protective gloves/protective clothing/eye protection/face protection
Use only outdoors or in a well-ventilated area
Keep only in original container
Response
Immediately call a POISON CENTER or doctor/physician
Inhalation
IF INHALED: Remove victim to fresh air and keep at rest in a position comfortable for breathing
Skin
IF ON SKIN (or hair): Take off immediately all contaminated clothing. Rinse skin with water/shower
Wash contaminated clothing before reuse
Eyes
IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing
Ingestion
IF SWALLOWED: Rinse mouth. DO NOT induce vomiting
Spills
Absorb spillage to prevent material damage
Storage
Store locked up
Store in a well-ventilated place. Keep container tightly closed
Store in corrosive resistant polypropylene container with a resistant inliner
Store in a dry place
Disposal
Dispose of contents/container to an approved waste disposal plant
Hazards not otherwise classified (HNOC)
None identified

3. Composition / information on ingredients

Component	CAS-No	Weight %
Water	7732-18-5	>78
Hydrochloric acid	7647-01-0	22

4. First-aid measures

General Advice If symptoms persist, call a physician.

Eye Contact Rinse immediately with plenty of water, also under the eyelids, for at least 15 minutes. Immediate medical attention is required.

Skin Contact Wash off immediately with plenty of water for at least 15 minutes. Immediate medical attention is required.

Inhalation Move to fresh air. If breathing is difficult, give oxygen. Do not use mouth-to-mouth method if victim ingested or inhaled the substance; give artificial respiration with the aid of a pocket mask equipped with a one-way valve or other proper respiratory medical device. Immediate

medical attention is required.

Ingestion Do not induce vomiting. Call a physician or Poison Control Center immediately.

Most important symptoms/effects Causes burns by all exposure routes. Product is a corrosive material. Use of gastric lavage or emesis is contraindicated. Possible perforation of stomach or esophagus should be investigated: Ingestion causes severe swelling, severe damage to the delicate tissue and danger of perforation

Notes to Physician Treat symptomatically

5. Fire-fighting measures

Suitable Extinguishing Media Substance is nonflammable; use agent most appropriate to extinguish surrounding fire.

Unsuitable Extinguishing Media No information available

Flash Point No information available
Method - No information available

Autoignition Temperature No information available
Explosion Limits
Upper No data available
Lower No data available
Sensitivity to Mechanical Impact No information available
Sensitivity to Static Discharge No information available

Specific Hazards Arising from the Chemical
Non-combustible, substance itself does not burn but may decompose upon heating to produce corrosive and/or toxic fumes.

Hazardous Combustion Products
Hydrogen chloride gas Carbon monoxide (CO) Carbon dioxide (CO_2) Hydrogen
Protective Equipment and Precautions for Firefighters
As in any fire, wear self-contained breathing apparatus pressure-demand, MSHA/NIOSH (approved or equivalent) and full protective gear.

NFPA

Health	Flammability	Instability	Physical hazards
3	0	1	N/A

6. Accidental release measures

Personal Precautions Use personal protective equipment. Ensure adequate ventilation. Evacuate personnel to safe areas.

Environmental Precautions Should not be released into the environment. See Section 12 for additional ecological information.

Methods for Containment and Clean Up Soak up with inert absorbent material. Keep in suitable, closed containers for disposal.

7. Handling and storage

Handling Use only under a chemical fume hood. Ensure adequate ventilation. Wear personal protective equipment. Do not get in eyes, on skin, or on clothing. Do not breathe vapors or spray mist. Do not ingest.

Storage Keep containers tightly closed in a dry, cool and well-ventilated place.

8. Exposure controls / personal protection

Exposure Guidelines

Component	ACGIH TLV	OSHA PEL	NIOSH IDLH
Hydrochloric acid	Ceiling: 2 ppm	Ceiling: 5 ppm Ceiling: 7 mg/m³ (Vacated) Ceiling: 5 ppm (Vacated) Ceiling: 7 mg/m³	IDLH: 50 ppm Ceiling: 5 ppm Ceiling: 7 mg/m³

Component	Quebec	Mexico OEL (TWA)	Ontario TWAEV
Hydrochloric acid	Ceiling: 5 ppm Ceiling: 7.5 mg/m³	Ceiling: 5 ppm Ceiling: 7 mg/m³	CEV: 2 ppm

Legend

ACGIH *- American Conference of Governmental Industrial Hygienists*
OSHA *- Occupational Safety and Health Administration*
NIOSH IDLH: *The National Institute for Occupational Safety and Health Immediately Dangerous to Life or Health*

Engineering Measures Use only under a chemical fume hood. Ensure that eyewash stations and safety showers are close to the workstation location.

Personal Protective Equipment

Eye/face Protection Wear appropriate protective eyeglasses or chemical safety goggles as described by OSHA's eye and face protection regulations in 29 CFR 1910.133 or European Standard EN166.

Skin and body protection Wear appropriate protective gloves and clothing to prevent skin exposure.

Respiratory Protection Follow the OSHA respirator regulations found in 29 CFR 1910.134 or European Standard EN 149. Use a NIOSH/MSHA or European Standard EN 149 approved respirator if exposure limits are exceeded or if irritation or other symptoms are experienced.

Hygiene Measures Handle in accordance with good industrial hygiene and safety practice.

9. Physical and chemical properties

Physical State	Liquid
Appearance	Clear
Odor	pungent
Odor Threshold	No information available
pH	1
Melting Point/Range	-74 °C / -101.2 °F
Boiling Point/Range	81.5 - 110 °C / 178.7 230 °F @ 760 mmHg
Flash Point	No information available
Evaporation Rate	> 1.00 (Butyl Acetate = 1.0)
Flammability (solid,gas)	Not applicable
Flammability or explosive limits	
Upper	No data available
Lower	No data available
Vapor Pressure	5.7 mmHg @ 0 °C
Vapor Density	1.26
Specific Gravity	1.0 - 1.2
Solubility	Miscible with water
Partition coefficient; n-octanol/water	No data available
Autoignition Temperature	No information available
Decomposition Temperature	No information available
Viscosity	No information available

10. Stability and reactivity

Reactive Hazard None known, based on information available

Stability Stable under normal conditions. Water reactive.

Conditions to Avoid Incompatible products. Excess heat. Exposure to moist air or water.

Incompatible Materials Metals, Oxidizing agents, Reducing agents, Acids, Bases, Aldehydes

Hazardous Decomposition Products Hydrogen chloride gas, Carbon monoxide (CO), Carbon dioxide (CO_2), Hydrogen

Hazardous Polymerization Hazardous polymerization does not occur.

Hazardous Reactions May react with metals and lead to the formation of flammable hydrogen gas. Corrosive to metals.

11. Toxicological information

Acute Toxicity

Product Information

Oral LD50 Based on ATE data, the classification criteria are not met. ATE > 2000 mg/kg.

Dermal LD50 Based on ATE data, the classification criteria are not met. ATE > 2000 mg/kg.

Vapor LC50 Based on ATE data, the classification criteria are not met. ATE > 20 mg/l.

Component Information

Component	LD50 Oral	LD50 Dermal	LC50 Inhalation
Water	-	Not listed	Not listed
Hydrochloric acid	LD50 238 - 277 mg/kg (Rat)	LD50 > 5010 mg/kg (Rabbit)	LC50 = 1.68 mg/L (Rat) 1 h

Toxicologically Synergistic Products No information available

Delayed and immediate effects as well as chronic effects from short and long-term exposure

Irritation Causes burns by all exposure routes

Sensitization No information available

Carcinogenicity The table below indicates whether each agency has listed any ingredient as a carcinogen.

Component	CAS-No	IARC	NTP	ACGIH	OSHA	Mexico
Water	7732-18-5	Not listed	Not listed	Not listed	Not listed	Not listed
Hydrochloric acid	7647-01-0	Not listed	Not listed	Not listed	Not listed	Not listed

Mutagenic Effects No information available

Reproductive Effects No information available.

Developmental Effects No information available.

Teratogenicity No information available.

STOT - single exposure Respiratory system

STOT - repeated exposure None known

Aspiration hazard No information available

Symptoms / effects,both acute and delayed Product is a corrosive material. Use of gastric lavage or emesis is contraindicated. Possible perforation of stomach or esophagus should be investigated: Ingestion causes severe swelling, severe damage to the delicate tissue and danger of perforation

Endocrine Disruptor Information No information available

Other Adverse Effects The toxicological properties have not been fully investigated.

12. Ecological information

Ecotoxicity

Do not empty into drains.

Component	Freshwater Algae	Freshwater Fish	Microtox	Water Flea
Hydrochloric acid	-	282 mg/L LC50 96 h	-	-

Persistence and Degradability Persistence is unlikely based on information available.

Bioaccumulation/ Accumulation No information available.

Mobility No information available.

13. Disposal considerations

Waste Disposal Methods Chemical waste generators must determine whether a discarded chemical is classified as a hazardous waste. Chemical waste generators must also consult local, regional, and national hazardous waste regulations to ensure complete and accurate classification.

14. Transport information

DOT

UN-No UN1789
Proper Shipping Name HYDROCHLORIC ACID SOLUTION
Hazard Class 8
Packing Group II

TDG

UN-No UN1789
Proper Shipping Name HYDROCHLORIC ACID SOLUTION
Hazard Class 8
Packing Group II

IATA

UN-No UN1789
Proper Shipping Name HYDROCHLORIC ACID SOLUTION
Hazard Class 8
Packing Group II

IMDG/IMO

UN-No UN1789
Proper Shipping Name HYDROCHLORIC ACID, SOLUTION
Hazard Class 8
Packing Group II

15. Regulatory information

International Inventories

Component	TSCA	DSL	NDSL	EINECS	ELINCS	NLP	PICCS	ENCS	AICS	IECSC	KECL
Water	X	X	-	231-791-2	-		X	-	X	X	X
Hydrochloric acid	X	X	-	231-595-7	-		X	X	X	X	X

Legend:
X - Listed
E - Indicates a substance that is the subject of a Section 5(e) Consent order under TSCA.
F - Indicates a substance that is the subject of a Section 5(f) Rule under TSCA.
N - Indicates a polymeric substance containing no free-radical initiator in its inventory name but is considered to cover the designated polymer made with any free-radical initiator regardless of the amount used.
P - Indicates a commenced PMN substance
R - Indicates a substance that is the subject of a Section 6 risk management rule under TSCA.
S - Indicates a substance that is identified in a proposed or final Significant New Use Rule
T - Indicates a substance that is the subject of a Section 4 test rule under TSCA.
XU - Indicates a substance exempt from reporting under the Inventory Update Rule, i.e. Partial Updating of the TSCA Inventory Data Base Production and Site Reports (40 CFR 710(B).
Y1 - Indicates an exempt polymer that has a number-average molecular weight of 1,000 or greater.
Y2 - Indicates an exempt polymer that is a polyester and is made only from reactants included in a specified list of low concern reactants that comprises one of the eligibility criteria for the exemption rule.

U.S. Federal Regulations

TSCA 12(b) Not applicable

SARA 313

Component	CAS-No	Weight %	SARA 313 - Threshold Values %
Hydrochloric acid	7647-01-0	22	1.0

SARA 311/312 Hazard Categories

Acute Health Hazard	Yes
Chronic Health Hazard	No
Fire Hazard	No
Sudden Release of Pressure Hazard	No
Reactive Hazard	No

CWA (Clean Water Act)

Component	CWA - Hazardous Substances	CWA - Reportable Quantities	CWA - Toxic Pollutants	CWA - Priority Pollutants
Hydrochloric acid	X	5000 lb	-	-

Clean Air Act

Component	HAPS Data	Class 1 Ozone Depletors	Class 2 Ozone Depletors
Hydrochloric acid	X		-

OSHA Occupational Safety and Health Administration
Not applicable

Component	Specifically Regulated Chemicals	Highly Hazardous Chemicals
Hydrochloric acid	-	TQ: 5000 lb

CERCLA

Component	Hazardous Substances RQs	CERCLA EHS RQs
Hydrochloric acid	5000 lb	5000 lb

California Proposition 65 This product does not contain any Proposition 65 chemicals

U.S. State Right-to-Know Regulations

Component	Massachusetts	New Jersey	Pennsylvania	Illinois	Rhode Island
Water	-	-	X	-	-
Hydrochloric acid	X	X	X	X	X

U.S. Department of Transportation

Reportable Quantity (RQ):	N
DOT Marine Pollutant	N
DOT Severe Marine Pollutant	N

U.S. Department of Homeland Security
This product contains the following DHS chemicals:

Component	DHS Chemical Facility Anti-Terrorism Standard
Hydrochloric acid	0 lb STQ (anhydrous); 11250 lb STQ (37% concentration or greater)

Other International Regulations

Mexico - Grade No information available

Canada
This product has been classified in accordance with the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all the information required by the CPR

WHMIS Hazard Class E Corrosive material

16. Other information

Prepared By
Regulatory Affairs
Thermo Fisher Scientific
Email: EMSDS.RA@thermofisher.com

Creation Date 24-Aug-2009
Revision Date 24-Feb-2014
Print Date 24-Feb-2014
Revision Summary This document has been updated to comply with the US OSHA HazCom 2012 Standard replacing the current legislation under 29 CFR 1910.1200 to align with the Globally Harmonized System of Classification and Labeling of Chemicals (GHS)

Disclaimer
The information provided in this Safety Data Sheet is correct to the best of our knowledge, information and belief at the date of its publication. The information given is designed only as a guidance for safe handling, use, processing, storage, transportation, disposal and release and is not to be considered a warranty or quality specification. The information relates only to the specific material designated and may not be valid for such material used in combination with any other materials or in any process, unless specified in the text

End of SDS

Hazardous Chemicals

Hazardous Chemicals

The laboratory is a chemical use area for potentially hazardous compounds. The following are the hazard classes of chemicals used in this course and for which this laboratory is designated as a use area:

1. **Carcinogens**
2. **Corrosives**
3. **Flammable and combustible solids and liquids**
4. **Reproductive Toxins**

Hazardous Waste

Cation Metal Waste: Label is **WHITE** and is used in all CHEM 2 courses.

DO NOT FILL ABOVE THIS LINE

HAZARDOUS WASTE

Chem 2 Experiment

Cation Metal Waste

Follow these instructions:

- **Cap lid when not in use**
- **Leave in fume hood**
- **Call Dispensary when full**

FOR WASTE ONLY	Chemical Waste Composition: Aluminum, Bismuth, Chromium, Cobalt, Copper, Iron, Lead, Manganese, Silver, Zinc,

DANGER

Dithizone in Chloroform Waste: Label is **BLUE** and is used only in CHEM 2C.

KEEP BOTTLE UNCAPPED
DO NOT REMOVE FROM FUMEHOOD
CALL DISPENSARY WHEN FULL

HAZARDOUS WASTE

Chem 2C Experiment

Qualitative Analysis

Chemical Waste Composition:

Chloroform, Acetone,
Dithizone,

WASTE ONLY

DO NOT LEAVE BOTTLE UNCAPPED

-Flammable-
Toxic

Statistical Treatment of Data

Every measurement made in the laboratory is subject to error. Although you should try to minimize error, two types of errors will occur. Systematic or Determinate Errors are those errors which are reproducible and which can be corrected. Examples are errors due to a miscalibrated piece of glassware or a balance that consistently weighs light. Random or Indeterminate Errors are due to limitations of measurement that are beyond the experimenter's control. These errors cannot be eliminated and lead to both positive and negative fluctuations in successive measurements. Examples are a difference in readings by different observers, or the fluctuations in equipment due to electrical noise.

You will be graded by your ability to obtain accurate results. Accuracy describes how close your result is to the true value. Another related term is precision. Precision describes how close your results from different trials are to each other. Data of high precision indicates small random errors and leads experimenters to have confidence in their results. Data that is highly accurate suggests that there is little systematic error. A well-designed experiment (and a well-trained experimenter) should yield data that is both precise and accurate.

In an effort to describe and quantify the random errors which will occur during the course of the Chemistry 2 laboratory you will be asked to report an average, a standard deviation, a 90% confidence limit, and a relative deviation. You may also have to analyze multiple trials to decide whether or not a certain piece of data should be discarded. The following sections describe these procedures.

1. Average and Standard Deviation

The average or mean, $\bar{x}$, is defined by

$$\bar{x} = \frac{\sum x_i}{n}$$

where each x_i is one measurement and n is the number of trials.

The standard deviation, s, measures how close values are clustered about the mean. The standard deviation for small samples is defined by

$$s = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n - 1}}$$

The smaller the value of s, the more closely packed the data is about the mean—or, in other words, the measurements are more precise.

2. Confidence Limits

In general chemistry with a relatively small number of trials, we use a *t-distribution* (also called *Student t-distribution)* for a population mean estimation.

The *t-statistic* is determined by

$$t = \frac{\overline{x} - \mu}{\frac{s}{\sqrt{n}}}$$

where $\bar{x}$ is the sample mean, μ is the population mean, s is the standard deviation, and n is the sample size. The *t-statistic* distribution is called the *t-distribution*. The *t-distribution* approximates the normal distribution curve as the sample size increases (n).

The particular *t-distribution* is determined by the number of degrees of freedom. For the purposes of estimating the mean from a sample in the general chemistry experiments, the degree of freedom is calculated as the number of independent trials minus one. Then, the *t-distribution* determined by the specified *n - 1* degrees of freedom represents the sample mean distribution with respect to the true mean divided by $\frac{s}{\sqrt{n}}$. Using this information, an experimenter can formulate a confidence limit for that mean.

Confidence limits provide an indication of data precision. For example, a 90% confidence limit of ±2.0 indicates that there is a 90% probability that the true average of an infinite collection of data is within ±2.0 of the calculated average of a limited collection. Clearly, the more precise a set of data, the smaller the confidence interval. Thus, a small confidence interval is always the goal of any experiment. In General Chemistry, you will be required to calculate the 90% confidence interval for all experimental collections of data. The formula to do this is:

$$Confidence\ Limit = (t_{critical})\left(\frac{s}{\sqrt{n}}\right)$$

where s is the standard deviation, n is the number of trials, and t_{critical} is the critical value in a *t-distribution* table in statistics. A small section of the *t-distribution* table is shown at the end of this section. For the calculation of 90% confidence limits in General Chemistry, please use the following values:

Number of Trials (n)	$t_{critical}$
2	6.314
3	2.920
4	2.353
5	2.132
6	2.015

You should always report your result as the average ± the 90% confidence limit.

***t-distribution* table**

Confidence level / n	90%	95%	99%
2	6.314	12.71	63.66
3	2.920	4.303	9.925
4	2.353	3.182	5.841
5	2.132	2.776	4.604
6	2.015	2.571	4.032
∞	1.645	1.960	2.576

3. Relative Deviation

The relative average deviation, *d*, like the standard deviation, is useful to determine how data are clustered about a mean. The advantage of a relative deviation is that it incorporates the relative numerical magnitude of the average.

The relative average deviation, *d*, is calculated in the following way.

a.) Calculate the average, $\bar{x}$, with all data that are of high quality.

b.) Calculate the deviation, $|x_i - \overline{x}|$, of each good piece of data.

c.) Calculate the average of these deviations.

d.) Divide that average of the deviations by the mean of the good data.

This number is generally expressed as parts per thousand (ppt). You can do this by simply multiplying by 1000.

Please report the relative average deviation (ppt) in addition to the standard deviation in all experiments.

4. Analysis of Poor Data: Q-test

Sometimes a single piece of data is inconsistent with other data. You need a method to determine, or test, if the data in question is so poor that it should be excluded from your calculations. Many tests have been developed for this purpose. One of the most common is what is known as the Q-test. To determine if a data should be discarded by this test you first need to calculate the difference of the data in question from the data closest in value (this is called the "gap"). Next, you calculate the magnitude of the total spread of the data by calculating the difference between the data in question and the data furthest away in value (this is called the "range"). You will then calculate the Q_{Data}, given by

$$Q_{Data} = \frac{gap}{range}$$

and compare the value to that given in the table below. The values in the table below are given for the 90% confidence level. If the Q_{Data} is greater than the $Q_{Critical}$ then the data can be discarded with 90% confidence (the value has a less than 10% chance of being valid).

Number of Trials	$\mathbf{Q}_{Critical}$
3	0.94
4	0.76
5	0.64
6	0.56

While the Q test is very popular, it is not always useful for the small samples you will have (you will generally only do triplicate trials).

Keep in mind that you also always have the right to discard a piece of data that you are sure is of low quality. That is, when you are aware of a poor collection. However, beware of discarding data that do not meet the Q test. You may be discarding your most accurate determination!

An Introduction to Excel

In chemistry, as well as in other analytical sciences, it is important to not only know how to collect quality data, but also know how to analyze and manipulate that data to investigate your hypothesis. A spreadsheet program, such as Microsoft Excel, is an especially helpful tool to use for viewing and manipulating data, as it can be used to quickly perform complex calculations on large sets of data, as well as to rearrange raw data into easy to understand graphical representations.

In this guide, you will learn how to create a basic spreadsheet in Excel, and use formulas to quickly perform calculations on your data. You will also learn how to make graphs for your post-lab reports.

This guide uses Microsoft Excel 2016, which is available as a free download for students via:

► http://officedownload.ucdavis.edu

The above link can be accessed by logging in with your campus Kerberos (CAS) account. If you do not wish to download Microsoft Office onto your personal computer, Excel is also available for use at all of the computer labs on campus.

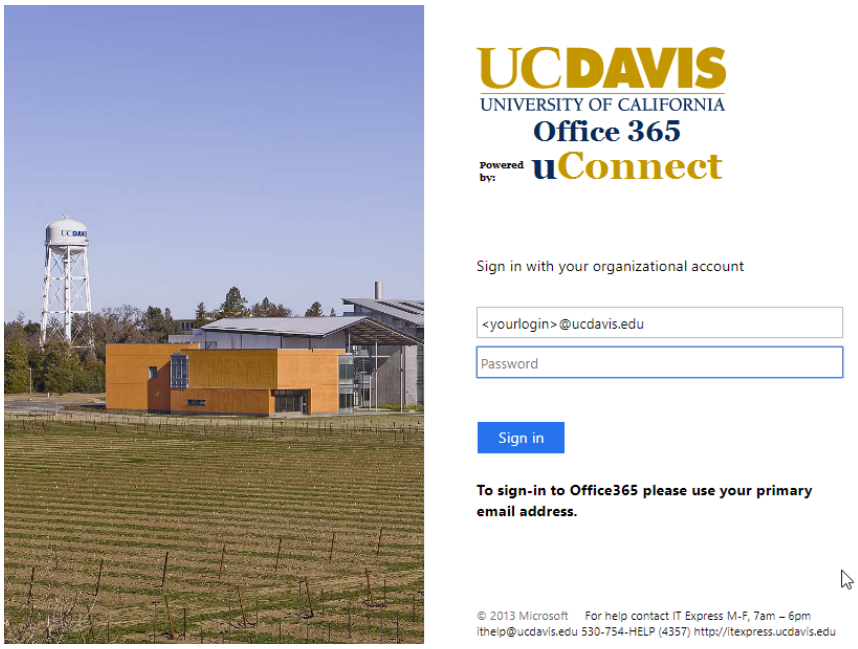


Figure 1. Use your UC Davis login information to access Microsoft Office 365.

Figure 2. You can install Microsoft Office 2016 by clicking on the "Install Office 2016" button once you've logged in.

Excel Basics

1. Open a new spreadsheet in Excel 2016. The image below shows a section of the blank worksheet.

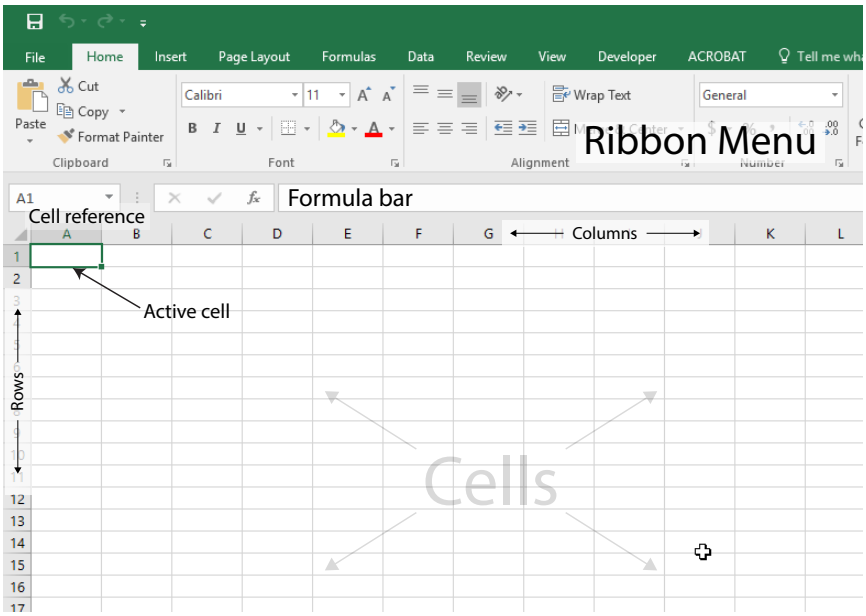


Figure 3. A blank spreadsheet in Excel 2016.

The gray rectangles that make up the spreadsheet are called **cells**, and the **active cell**, or the cell you are currently typing in, has a green outline around it with a handle at the bottom right.

Each cell has its own **cell reference** that consists of the letter of the column and the number of the row it is currently in. The cell reference is analogous to a variable in algebra, where the reference refers to the data inside of the cell. In the image above, the cell reference of the active cell is **A1**.

The **formula bar** displays the formulas in the active cell. If there are no formulas in the active cell, the formula bar displays the text in the cell.

The **ribbon menu** contains a variety of commands to edit and manipulate the data in the spreadsheet. In this guide, we will mainly be using the **Home** and **Insert** menus to edit our spreadsheet.

2. For this section of the guide, we will use sample data from the 2A experiment, *Volumetric Analysis*.

   Enter the data in columns, using one cell for each data point. Make sure all the data points from the same trial are in the same row.

   In this example, we also include a header row to help keep track of the data columns, although a header row is not required for the program to create graphs or perform calculations.

   As you can see in the image below, each row represents a separate trial for the experiment. Column B shows the mass of KHP used, and column C shows the volume of NaOH needed to reach the endpoint.

	A	B	C
1	Trial	Mass KHP (g)	Vol NaOH (mL)
2	1	0.31	16.25
3	2	0.32	15.6
4	3	0.35	16.3
5			

Figure 4. Sample data from 2A, *Volumetric Analysis*.

3. If we need to enter a series of **equal** intervals, such as a set of increasing wavelengths or time intervals, we can take advantage of Excel's auto-fill feature by using the **fill handle** at the bottom of an active area to quickly enter that series.

   Enter the first few values from your series. Then, click on the top-most cell containing data to make it the active cell. Hold the Shift key down and click on the bottom-most cell containing data to select the rest of the data points. The green outline will expand around the entire selected area.

   Hover your mouse cursor over the handle at the bottom right of the active area. The cursor will change into a small plus sign (+). Left-click and drag the handle down to another cell in the column to expand the green outline to that cell. A small hover box near the cursor also shows the value that cell will have once the series is expanded.

   Let go of the mouse button to fill the selected area with the expanded series. In the following image, notice how the series can be expanded from just two initial values.

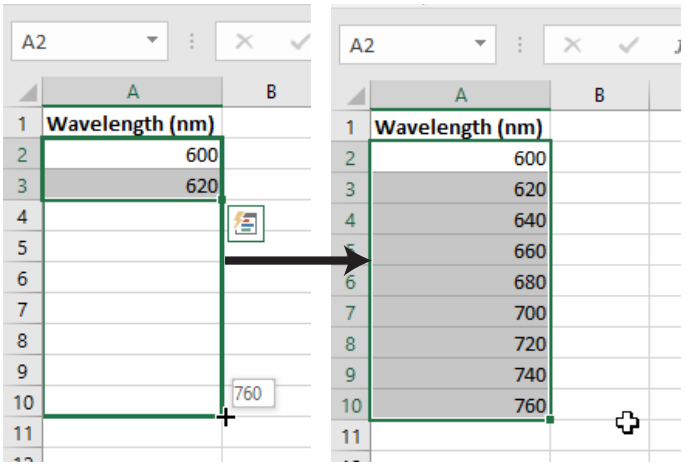


Figure 5. Using the Fill Handle to expand a series of increasing wavelengths.

The fill handle can be used across columns or rows, and can also be used to expand calculations, as you will see in the next section.

4. We may also want to change how many decimal places are displayed in each column or row, depending on what the experiment requires.

   To add or remove decimal places, select an area and right click anywhere in that area. Select **Format Cells...** from the context menu to bring up the Format Cells window.

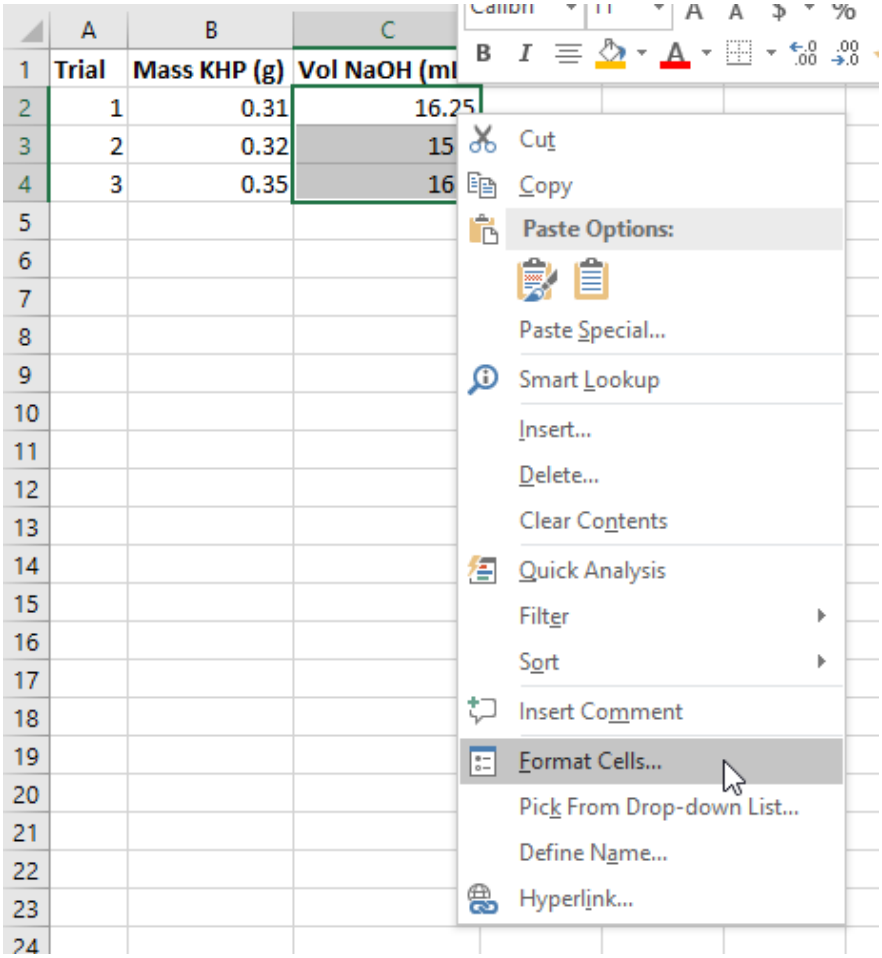


Figure 6. Select Format Cells... from the context menu.

The default category for a cell is **General**. Change the category to **Number** and set the number of decimal places as dictated by the experiment.

However, keep in mind that Excel **does not** allow you to set the number of significant figures, so you will still need to remember the rules for rounding significant figures in order to determine the number of decimal places to use.

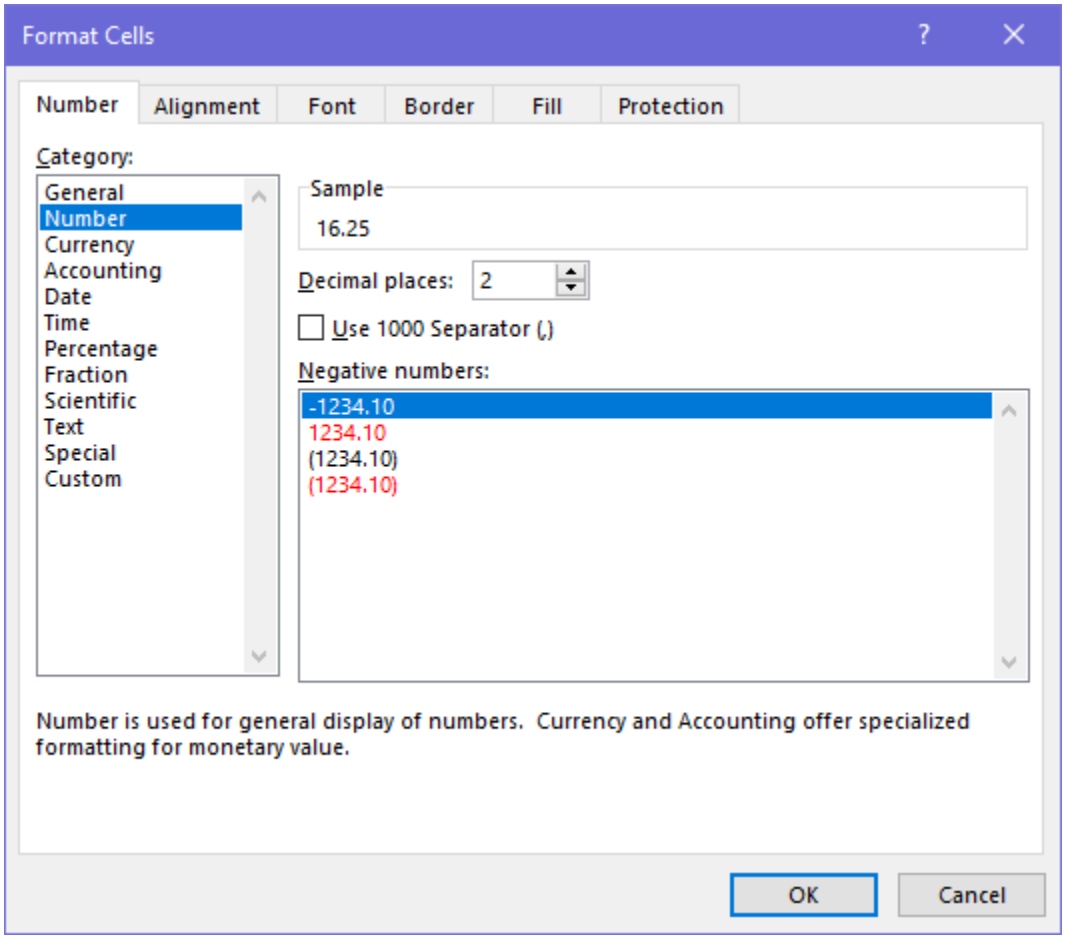


Figure 7. The Format Cells window showing the Number formatting.

Calculations in Excel

5. Now that we've entered our raw data, we can use Excel to quickly perform calculations with that data using **formulas**.

   Excel formulas always start with an equal sign (=). Formulas can use one or more *operators* or *functions*, and can contain a mix of *constants* and *cell references*. Note that Excel formulas using math operators follow the mathematical order of operations.

   Functions are a type of procedure you can perform in Excel, denoted with an equal sign (=), a function name, such as SUM or AVERAGE, and a set of parentheses containing one or more parameters separated by commas. There are many different functions in Excel, and you can press the *fx* button next to the Formula bar to view the full list. However, in the Chem 2 course, you will most likely only need to use use the mathematical functions listed below.

Common math functions for Excel	
=SUM(A1:A5)	Finds the sum of all the cells between cell A1 and cell A5.
=AVERAGE(A1:A5)	Finds the average of the values between cell A1 and cell A5.
=STDEV(A1:A5)	Finds the standard deviation of all the cells between cell A1 and cell A5.

   In the *Volumetric Analysis* experiment, we perform multiple titrations of KHP with NaOH to determine the molarity of an NaOH solution. We use the following stoichiometric equation to calculate the molarity of NaOH:

$$\frac{grams\ KHP\ \times \frac{1\ mol\ KHP}{204.2\ g\ KHP} \times \frac{1\ mol\ NaOH}{1\ mol\ KHP}}{volume\ NaOH\ added\ (L)} = Molarity\ (M) NaOH$$

   We can type this equation as an Excel formula using *cell references* to refer to the data we entered earlier. In this example, the mass of KHP is recorded in column B, and the volume of NaOH added is recorded in column C.

   Move to the next blank column in the spreadsheet and give it an appropriate header, such as **[NaOH] (M)**. In the row corresponding to the first trial, type out the formula using cell references to the data points from that trial. Trial 1 is recorded in *row 2*, so we refer to cells **B2** and **C2** in the formula.

   Be careful to follow the order of operations and use parentheses to group operations together if needed. Excel will highlight each cell being referenced in a different color, which you can use as a visual guide to double check that you are referring to the correct cells.

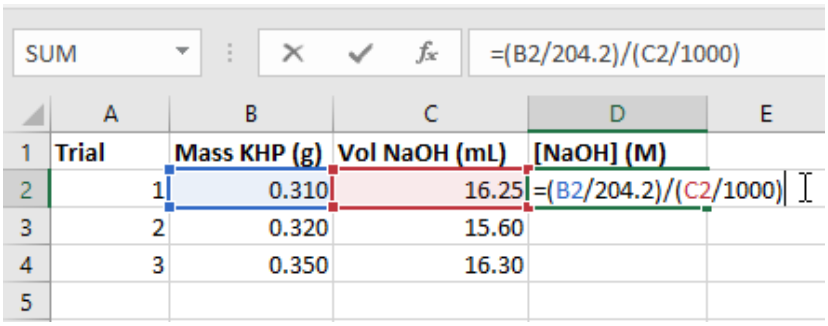


Figure 8. The equation typed into cell D2 as an Excel formula.

Hit the enter key, and the formula will switch to the calculated value. You can double click on the cell to show the formula again if you wish to make any edits.

Now, we can expand that formula to apply to the other rows in the spreadsheet. Click and drag the fill handle down to the bottom-most row of data.

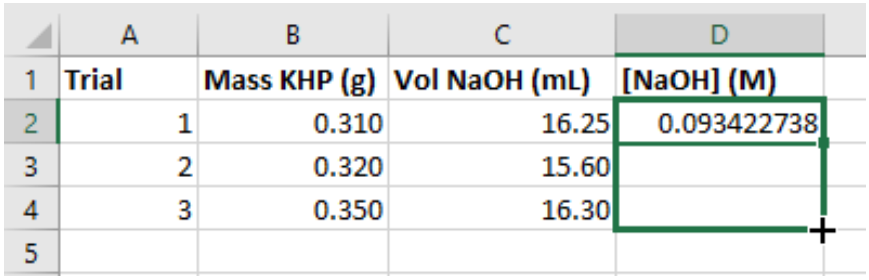


Figure 9. Click and drag the handle down to the last row of data.

Excel will automatically perform the calculation for every row in the selected area. Note how the cell references are updated for row 4 in the picture below.

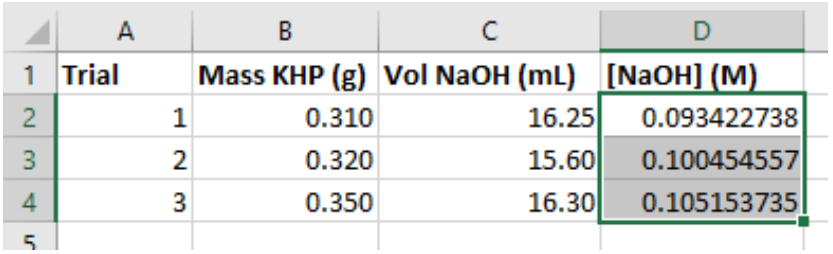


Figure 10. The formula bar showing the updated cell references.

When you have a large number of trials and you need to use multiple steps in your calculation, it may be easier to do your calculations in Excel rather than on a calculator, because you only need to enter the calculation once.

6. Now, we can use functions in other cells to find the average, standard deviation, and so on. The image below shows the average for each of the 3 columns, again starting from cell B6 and using the fill handle to expand the formula across the 3 columns in row 6.

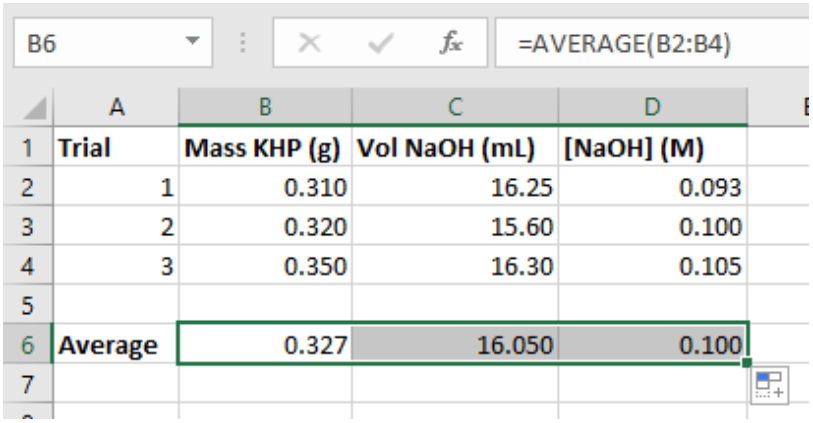


Figure 11. The formula bar shows the formula used to calculate the value in the cell.

Graphing in Excel

7. Excel is also useful for making graphical representations of data. Graphs are an extremely valuable tool in data analysis, because they depict the relationships between data points in a format that is easy to view at a glance.

   For this section of the guide, we will use the sample data found at the end of the *Strong Acid - Strong Base Titration* experiment to create a titration curve.

   Enter the data in 2 columns, and click on the top leftmost cell containing data. Then, while holding down shift, click on the bottom rightmost cell containing data to select the entire field of data. Then, go to the **Insert** tab of the ribbon menu to find the graphing options.

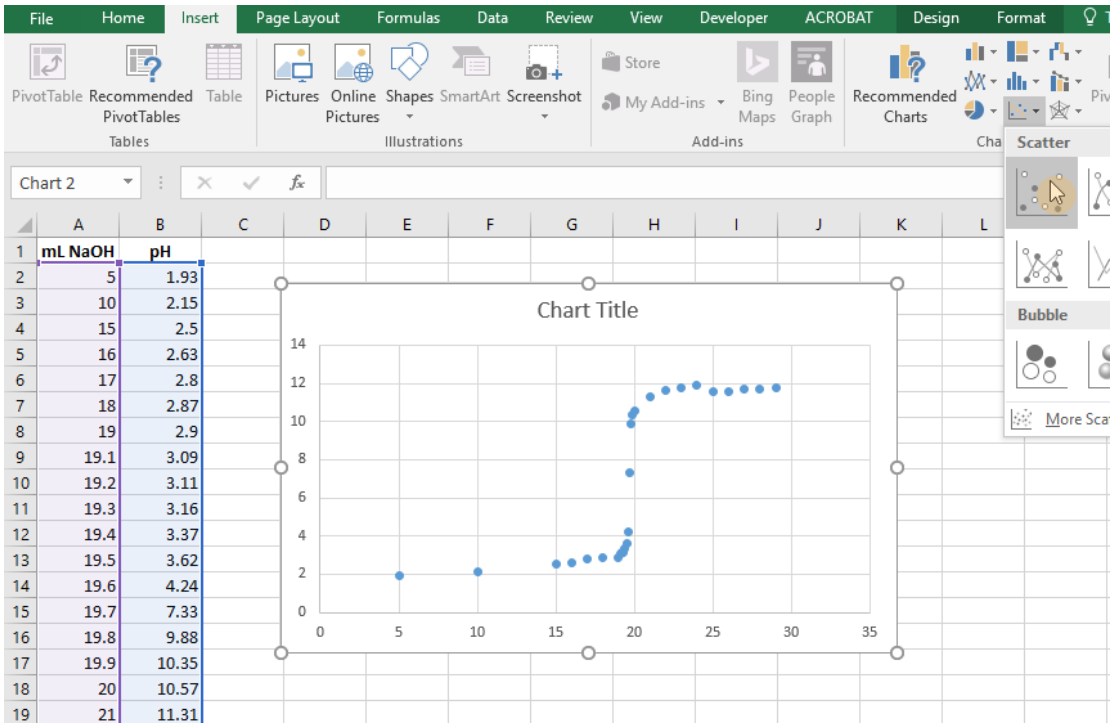


Figure 12. After selecting the data range, go to Insert > Scatter to plot the points on a graph.

   There are a variety of different graph types you can create in Excel. In General Chemistry, we will most commonly use the **scatter chart** to create graphs.

   With the data range selected, click on the **Insert Scatter (X, Y) Chart button** to plot the points on an xy-axis. This inserts a basic scatter graph into your spreadsheet, but we will want to edit the graph to add more information, such as axes labels or connecting lines.

8. First, let's add some lines to connect the data points and create the titration curve.

   You can open up the options menu for the data points by right clicking on any one of the points and clicking on **Format Data Series** from the context menu. A menu will pop up on the right side of the screen.

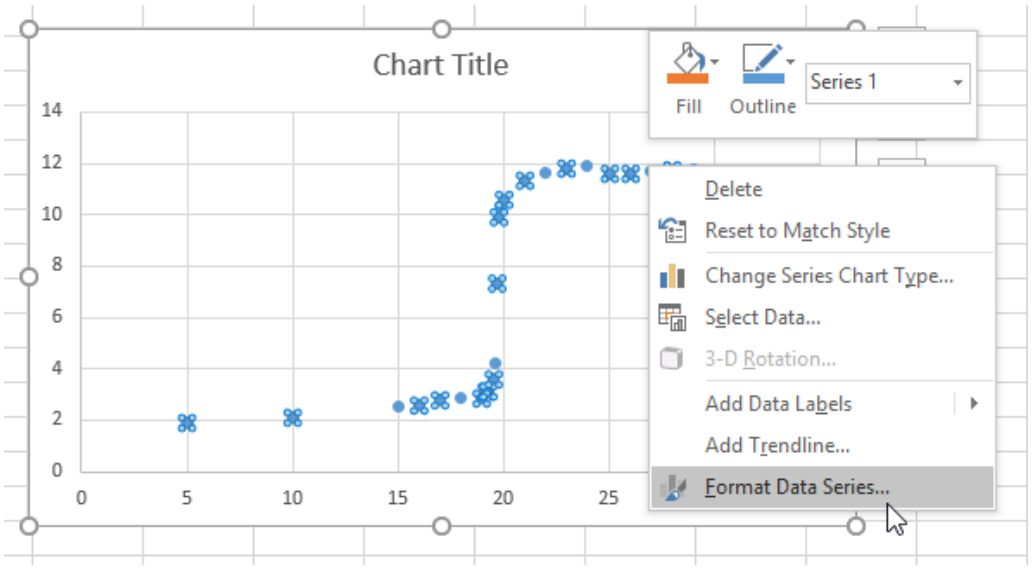


Figure 13. Select Format Data Series from the context menu to access more options.

In the Format Data Series menu, there are options to edit the Line and Marker appearances. You may have to click on the menu text to reveal all of the options.

To add lines between the points, click on the bubble next to **Solid line**.

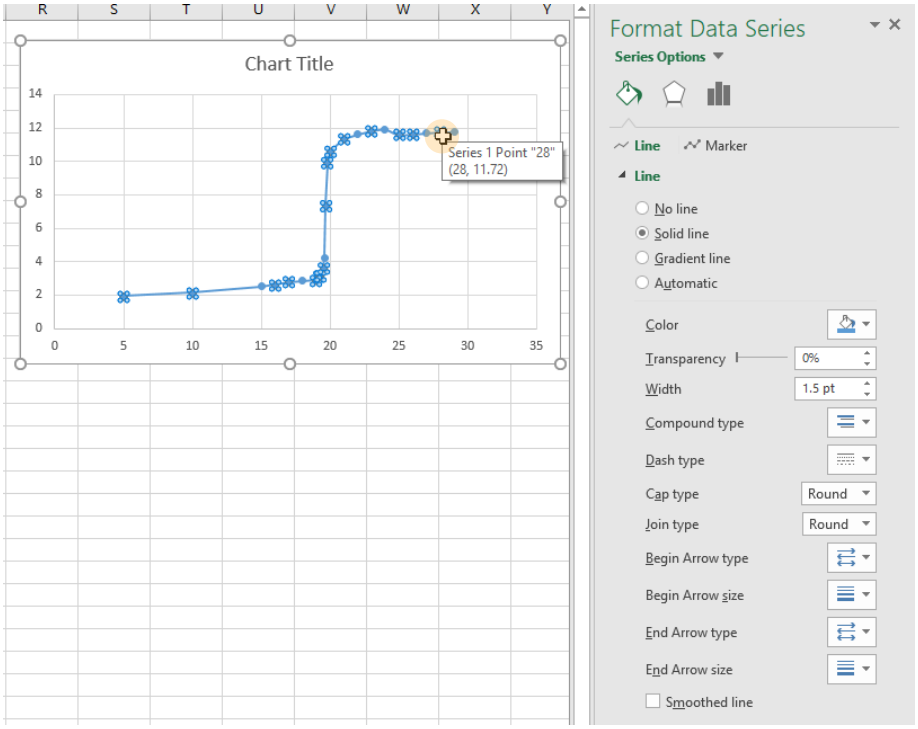


Figure 14. The titration curve with connecting lines added.

9. Next, we want to add descriptive labels to the x- and y- axes so others viewing the graph can understand what each axis represents. Select any part of the graph and click on the **+** button to insert chart elements. Check the box next to **Axis Titles** to insert text fields you can edit next to the x- and y- axes.

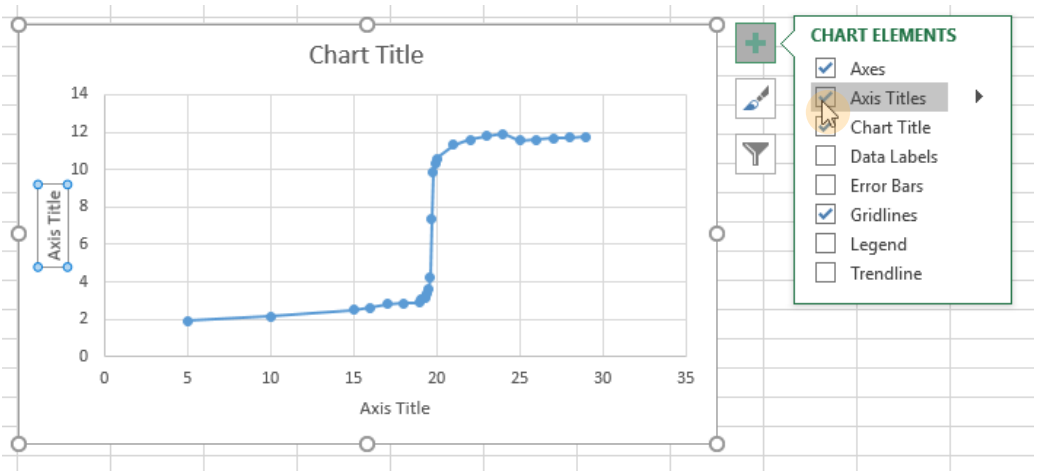


Figure 15. The Chart Elements menu.

Double click on each of the text fields to enable editing. Be sure to include your units in the axis titles, and don't forget to give your graph a descriptive title as well.

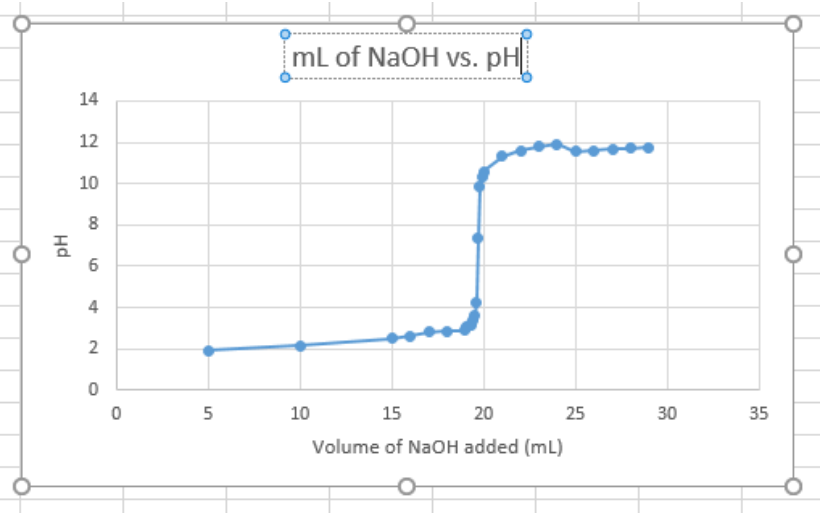


Figure 16. The titration curve with a title and axis labels added.

10. Finally, we can optionally change the range of each axis to minimize the amount of empty space on the graph. Right click on either axis and click on Format Axis to bring up the **Format Axis** options menu. Here, you can change the bounds on the axis to your liking.

    On this graph, there are no data points between 0 and 5, and 30 and 35 on the x-axis, so we will change the bounds to 5 and 30. The graph will automatically change to fit the new bounds.

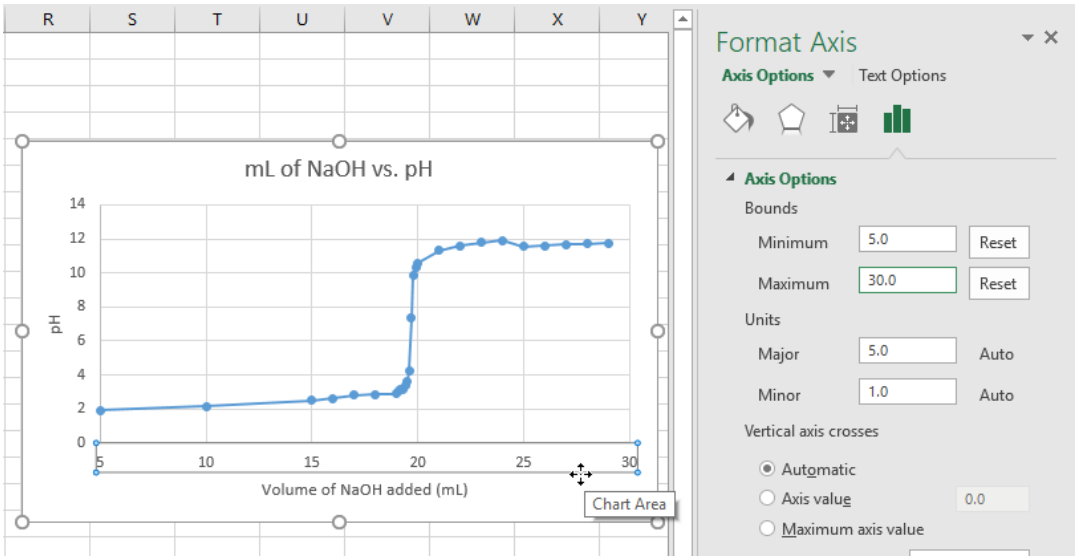


Figure 17. Changing the minimum and maximum bounds of the x-axis.

Common Laboratory Procedures

Handling Solids

1. General Guidelines for Handling Solids

1. Use a **clean** spatula or scoopula to transfer solid from bottles. Never use a contaminated spatula.
2. Never return unused solid to the reagent bottle. To eliminate waste, avoid removing more solid from a bottle than is necessary.
3. Never discard chemicals in the trashcan. Follow waste disposal procedures outlined in the Laboratory Manual.

2. Quantitative Transfer

Quantitative transfer refers to the moving of *all the contents* to be transferred from one container to another. Below is an illustration of how to properly weigh and transfer a solid using weighing paper. You will be using weighing boats rather than weighing paper, but the procedure is essentially the same.

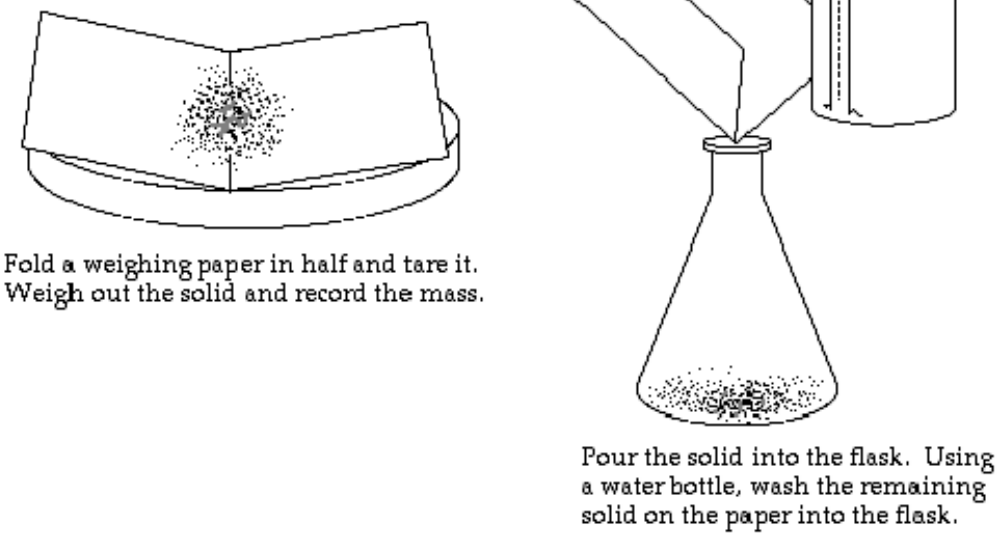


Figure 1. Quantitative Transfer of Solids

3. Using the Desiccator

You will occasionally be asked to use the desiccator during the laboratory course to dry some reagents. The desiccator contains some amount of desiccant, which absorbs moisture from air.

a. **Keep the desiccator closed at all times.**

   The desiccant will absorb moisture in the air extremely rapidly.

b. **Keep the desiccator tightly sealed with some vacuum grease.**

   To apply vacuum grease, put a pea-sized amount of grease on a paper towel and wipe it along the rim of the transparent cover. Make sure you do not use too much grease. Place the cover on top of the base and twist the cover 30 degrees to ensure a tight seal.

Desiccator Care

In the Chemistry 2 lab, we use Calcium Chloride as the desiccant. If water is found in the desiccator, discard the desiccant in the sink and rinse with copious amount of water until all solids are dissolved. Wipe the desiccator dry with a paper towel. Make sure all traces of water are removed before refilling from the 10 kg bucket of Calcium Chloride in your lab.

Hard to Open Desiccator

Do not try to force open a desiccator. You may accidentally shatter the glassware stored inside. Use an aluminum scoopula as a wedge and push it slowly into the space between the covers.

Notice

- Always keep the desiccator upright and closed in your locker.
- Clean up Calcium Chloride spill immediately. Moisture will damage drawers.

Handling Liquids

1. Drawing Solutions from a Reagent Bottle

Most reagent bottles in your laboratory have a small test tube holder attached for a disposable (dispo) plastic pipette. To avoid cross-contamination, always use the assigned dispo pipette to draw solutions from the reagent bottle. Do not use your glass pipet with reagent bottles.

Caution

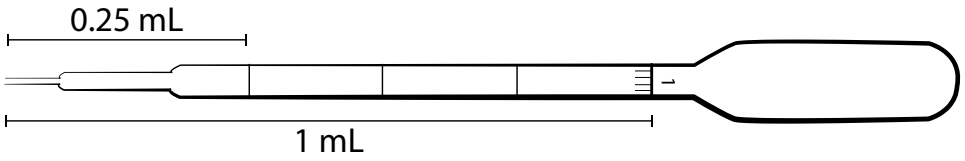
- Improper use of disposable pipets may cause serious injuries!
- Never point the pipet at yourself or others!
- Do not squeeze air into solutions with the dispo pipet. This may result in chemical splashes.
- Always put full dispo pipet in a test tube when carrying it to another part of the lab.

2. Estimating Volume with a Dispo Pipet

The dispo pipette may be used to transfer an estimated amount of solution. This is useful when working with non-limiting reagents or quickly making a solution that will be titrated later.

To draw 1mL of solution into an empty dispo pipet:

a. Squeeze the bulb to remove some air from the dispo pipet.
b. Submerge the tip of the dispo pipet in the solution.
c. Slowly release the pressure on the bulb and draw solution to the 1mL mark.
d. Without releasing pressure on the bulb, steadily remove the dispo pipet from the solution.



3. Transferring Liquid

a. When transferring liquids from a reagent bottle, always remove the cap/stopper and hold it in your hand. Never place the cap/stopper on the bench or contamination could result. Pour the liquid slowly and carefully to avoid spillage. You may find the use of a glass rod helpful, as shown below.

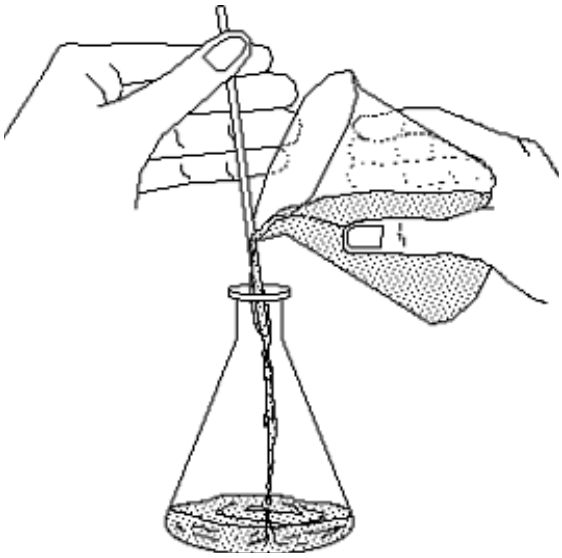


Figure 2. Liquid Transfer with Glass Stir Rod

b. With the exception of beakers, you should always use a funnel when transferring liquids from a container with a large opening to a container with a small opening.

4. Capping a Flask with Parafilm

During many experiments you will have to cap a flask to protect the contents from contamination. **Figure 3.** illustrates the proper method using Parafilm.

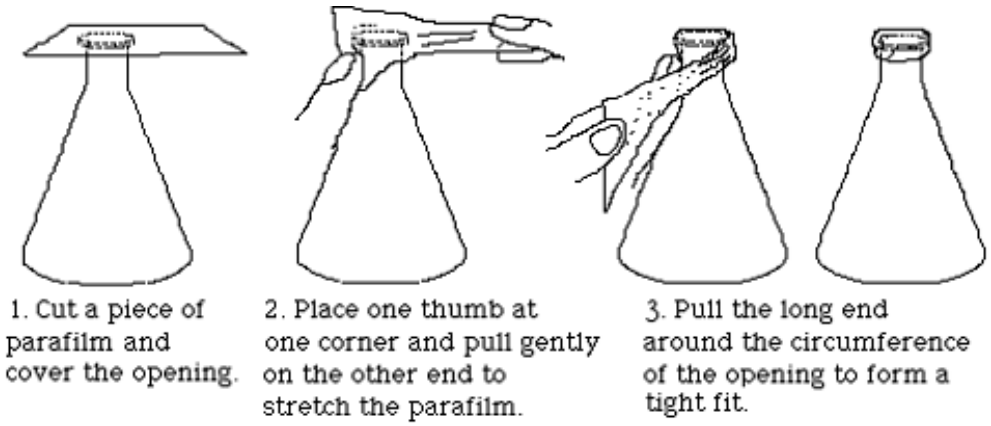


Figure 3. Capping A Flask

5. Measuring Liquid Volumes

Many glassware items have volume marks printed on them. Before using a piece of glassware to make a volume measurement, you should take a moment to study its calibrations to insure that you know how to read them properly.

- A beaker or Erlenmeyer flask can be used for rather rough measurements.
- A graduated cylinder can be used for measurements of moderate accuracy.
- A pipet is commonly used to transfer an accurately known volume of a liquid.

However, the accuracy of such a transfer is only as good as the technique of the operator will allow.

In making any volume measurement, the liquid level should always be the same as your eye level. Erlenmeyer flasks and graduated cylinders are usually filled/read by *raising them to your eye level* rather than by squatting down to bring your eye level to the bench top. The liquid level in a pipet is always lowered to the mark while the mark is held steady at eye level.

Burets: With practice, the position of the meniscus of a liquid in the 25 mL burets used in the Chemistry 2 labs can be estimated to within 0.02 mL.

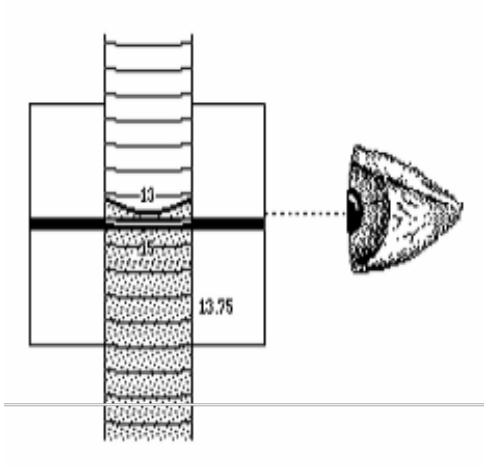


Figure 4. Reading the Meniscus

Figure 4. shows the use of a card with a dark strip on it to sharpen the image of the meniscus. You will find by experiment that if the top of the strip is positioned slightly below the level of the liquid in the buret, the bottom of the meniscus will be very easy to see.

Common Glassware in the Laboratory

Almost all of the glassware used in the Chemistry 2 laboratories are made with borosilicate glass, which is able to resist high temperatures and most chemicals.

1. Care and Maintenance of Laboratory Glassware

a. Always examine the glassware for chips and scratches before use. Damaged glassware may break during normal usage or cause bodily injuries.

b. Never place glassware near the edge of lab bench. Keep the work area clean and organized to prevent accidents and chemical spills.

c. Clean broken glassware must be disposed of inside the designated **Glass Disposal Box**. If box is full, ask the dispensary for a new one.

d. Clean all glassware with water. Make sure to rinse the glassware with DI water as a final step.

e. Never heat glassware to dryness. Add cold water with your **250 mL water squeeze bottle** when needed.

f. Never place a heated beaker in an ice bath, or vice versa. Allow the glassware to warm up or cool down gradually.

g. Never carry lab ware by the neck or cap. Always hold lab ware from the bottom and the side.

h. Never use tape or sticky labels on laboratory glassware. Always write on the white or blue label area with graphite pencil (a.k.a. “lead pencil”).

2. Beakers

Beakers can be used in the laboratory to estimate volume, storing liquids temporarily, and carry out certain reactions.

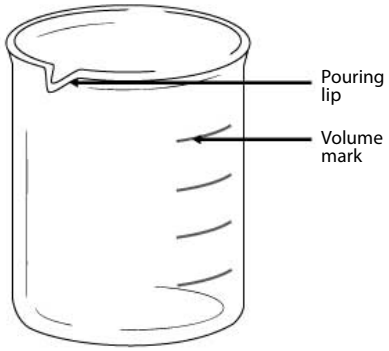
a. Always hold beakers from the bottom or the side. Never hold a beaker by the rim.

b. All beakers in the Chemistry 2 laboratories have a pouring lip to make pouring solutions easier.

c. All beakers in the Chemistry 2 laboratories have marks for estimating the volume. As noted on the glassware, there is a ±5% error for the largest volume mark.

d. Place a 100 mm watch glass on top of beaker when boiling water to speed up the process.

e. If needed, write labels in the frosted areas on the beaker with graphite pencils (a.k.a. "lead" pencils). **Do not use wax pencil or pen!**

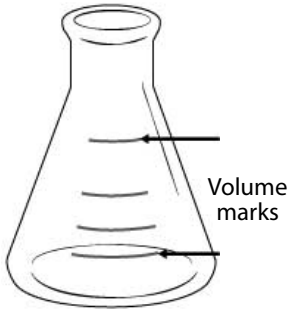


3. Erlenmeyer Flasks

Erlenmeyer flasks can be used in the laboratory to estimate volume, storing liquids temporarily, and carry out certain reactions.

a. All Erlenmeyer flasks in the Chemistry 2 laboratories have marks for estimating the volume. As noted on the glassware, there is a ±5% error for the largest volume mark.

b. If needed, write labels in the frosted areas on the beaker with graphite pencils (a.k.a. "lead" pencils). **Do not use wax pencil or pen!**

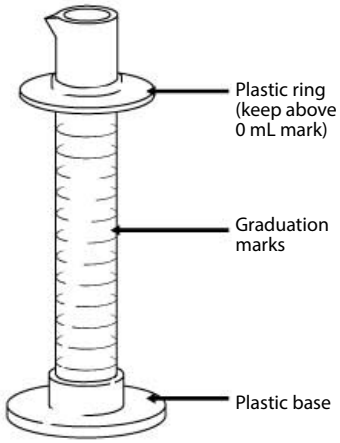


4. Graduated Cylinder

Graduated cylinders are used to measure a small volume of liquid with more precision than beakers and Erlenmeyer flasks.

a. The graduated cylinders in the Chemistry 2 laboratories include a plastic base and a plastic ring. The plastic ring is to protect the glass cylinder from shattering when the glassware is knocked over. Make sure the plastic ring is placed near the top of the cylinder.

b. To quickly measure out a specific amount of water, fill your **250 mL water squeeze bottle** with DI water and squeeze the desired amount of water into the graduated cylinder.



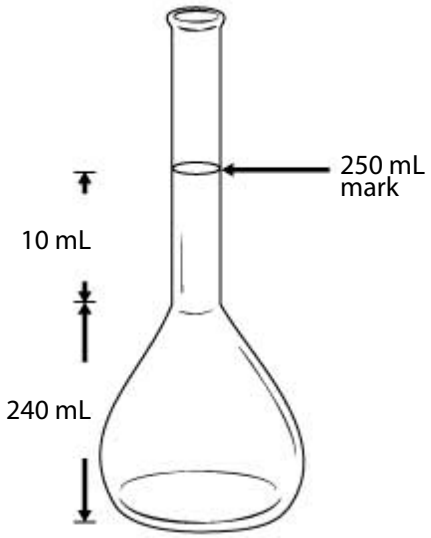
5. Volumetric Flasks

Volumetric flasks are very precisely calibrated glassware designed to contain one specific volume of liquid. You will only be allowed to have a limited number of volumetric flasks. If you need to make multiple solutions accurately with a volumetric flask, do not use multiple volumetric flasks. Instead, pour solutions you made in another container and reuse the same volumetric flask.

a. The 250 mL volumetric flask used in the Chemistry 2 laboratories has only one graduation mark for volume of 250 mL. As noted on the glassware, there is a ±0.12 mL error at 20 °C.

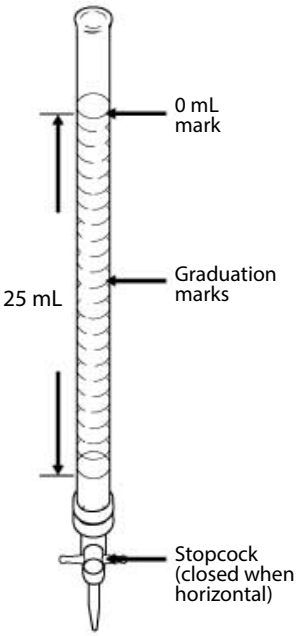
b. To fill a volumetric flask to the mark, quickly fill the flask to where the round base meets the neck. Cap the bottle and swirl or invert if needed. Then use a **250 mL water squeeze bottle** to fill to the volume mark. Notice that the volume between the neck and the 250 mL volume mark is only 10 mL.

c. Never use glass pipets or dispo pipets to draw solutions from volumetric flasks. Pipets will become stuck inside the flasks.



6. Burets

Burets are used to deliver a precise amount of solution. Unlike the volumetric flask and graduated cylinder, which are calibrated to measure the liquid contained in the glassware, burets are calibrated to measure the liquid delivered from the glassware. In the Chemistry 2 labs, the buret is mostly used for titrations.



a. Filling the buret:

- Always use a small beaker (100 mL or 150 mL) to transfer liquid into the buret. A funnel may be used to prevent spills as long as it is cleaned immediately after.
- Remove the buret from the buret clamp and hold it over a sink, below eye level.
- Check to make sure the stopcock is in the closed position.
- Hold the buret slightly below the 0mL mark with one hand. With your other hand, slowly pour the solution from the beaker into the buret. Stop before the liquid level reaches 0 mL
- If you used a funnel, place the funnel in the sink to clean.
- Replace the buret back in the buret clamp.

b. Cleaning the buret:

- To clean a buret, fill it to half way with DI water.
- At the sink, open the stopcock and drain out ~10 mL of water and close it. Then invert the buret and open the stopcock and drain out the rest from the top.

c. Conditioning the buret:

You should always condition your buret with your working solution before using it.

- Clean the buret with DI water.
- Fill the buret with 8-10 mL of the solution to be used. Open the stopcock to drain out a small amount from the tip into an appropriate waste container.
- Cap the top end with Parafilm. At the sink, hold the top of the buret between the thumb and finger of one hand, and hold the tip of the buret with another. Turn the buret horizontal and rotate the tip of the buret. Make sure all sides of the buret are washed with the solution.
- Pour the remaining solution in the buret into an appropriate waste container.

d. Dispensing solution from the buret:

- First, fill the buret with your solution to near the 0mL mark, but do not attempt to fill it to exactly 0.00 mL. Open the stopcock and drain out a very small amount to ensure no air bubbles exist in the tip. Record in your lab notebook your buret initial reading.
- Open the stopcock and drain the solution. Stop when the target volume is reached. Record the buret final reading in your lab notebook. The difference between the **initial reading** and the **final reading** is the volume dispensed.
- To dispense in small quantities, quickly turn the stopcock clockwise exactly 180 degrees. Repeat as needed.

7. Volumetric Pipet

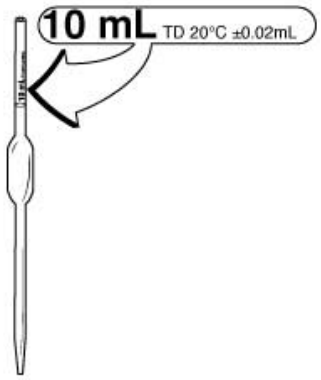
Similar to the buret, the volumetric pipet is designed to deliver a precise amount of solution.

a. The volume of liquid each pipet is designed to deliver is labeled on the glassware. Use the volumetric pipet *only* when you need to deliver the exact amount of solution with precision.

b. There is a bottle of volumetric pipet cleaning solution in each laboratory. Draw the cleaning solution into the pipet with a pipet bulb and dispel the solution

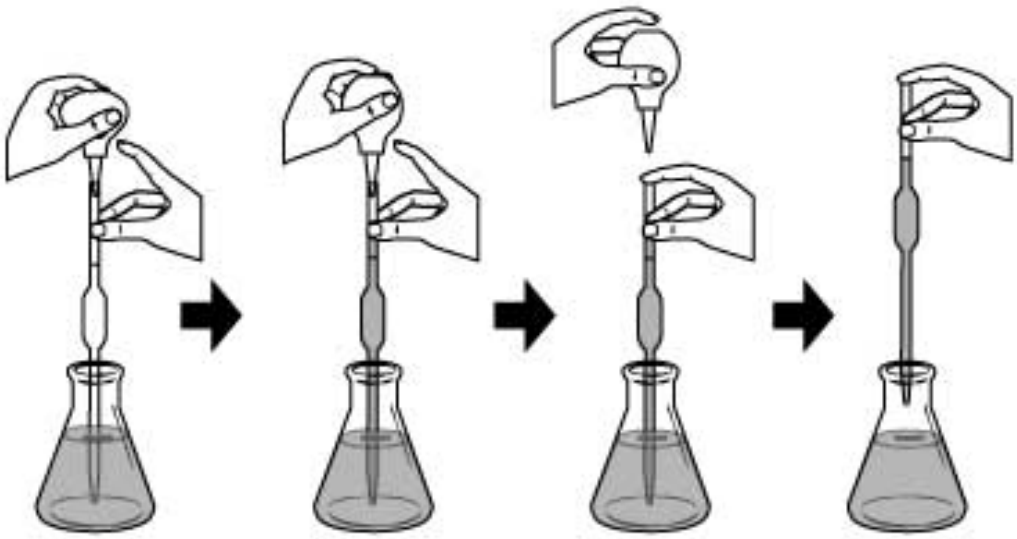
c. To condition a volumetric pipet, draw a small amount of your working solution into the pipet just above the volume mark. Drain the solution into an appropriate waste container.

d. Follow the illustration on the next page to learn how to use the volumetric pipet. You should practice using deionized water first to become proficient with the techniques.



Caution

- Never mouth pipet. Always use the pipet bulb with tip attached.
- Never point your pipet or pipet bulb at yourself or others.
- Never squeeze air into solutions as it may cause chemical splash.
- Never draw solutions into the bulb. Corrosive solutions will dissolve the rubber and contaminate the pipet.



1. To begin:
    - With one hand, hold the conditioned pipet vertical and the pointed end downward inside the container of your working solution. Place your other hand near the top of the pipet and keep the index finger free so that it can easily cap the pipet.
    - With your other hand, deflate the rubber pipet bulb with tip with your thumb.
    - Place the plastic pipet tip on the top of the pipet.
2. To draw the solution:
    - Slowly release your thumb and draw the liquid up the pipet and a few centimeters above the mark on the pipet. Keep the pipet submerged in solution to avoid drawing up air.
    - Lower the pipet so that it reaches the bottom of the container. Quickly remove the **pipet bulb with tip** and cap the pipet with your index finger.
3. To adjust the volume:
    - Raise the over-filled pipet. Raise the mark on the pipet to your eye level, tilt the receiver slightly, and touch the pointed tip of the pipet to a dry spot on its sidewall.
    - Rotate the pipet left and right slightly and let a small amount of air to enter the pipet and thereby allow the meniscus to fall exactly on the volume mark. Be patient, because if you overshoot the mark you must begin the whole process again.
4. To deliver the liquid:
    - Remove the accurately filled pipet from its container. Quickly dry the lower portion of the shaft with a single downward stroke of a laboratory tissue.
    - Tilt the final receiver slightly and while holding the pipet vertical, place its tip against the receiver wall so that when you take your finger off of the pipet mouth, liquid will flow smoothly down to the bottom of the vessel. Avoid splashing.
    - Do not squeeze solution out with the **pipet bulb with tip** and do not blow out the last drop. **The pipet is calibrated to deliver with one last drop left in the pipet.**

Using the Balance

A balance is used to measure the mass of an object. There are 4 balances assigned to your laboratory section for use in the adjoining balance room. These balances measure the mass to the nearest milligram. You will use these balances for most mass measurements in the Chemistry 2 lab experiments.

There is also a less precise "quick" balance in your lab room, between the fume hoods. You may use this balance to make rough measurements of non-limiting reagents quickly and speed up your experiment without compromising the experiment results.

1. On/Off Switching

a. To turn on the balance, remove all load from the weighing pan and press the **On** button.

b. To turn off the balance, press and hold the **Off** key for 2 seconds.

2. Simple Weighing

Open one of the draft shield sliding doors. Make sure the balance pan and surrounding area is clean. You can clean it with a balance brush or Kimwipe.

Next, shut the doors and press the **0/T button** to set the balance at zero.

Now, simply place the object to be weighed on the weighing pan and measure the mass to 0.001 grams.

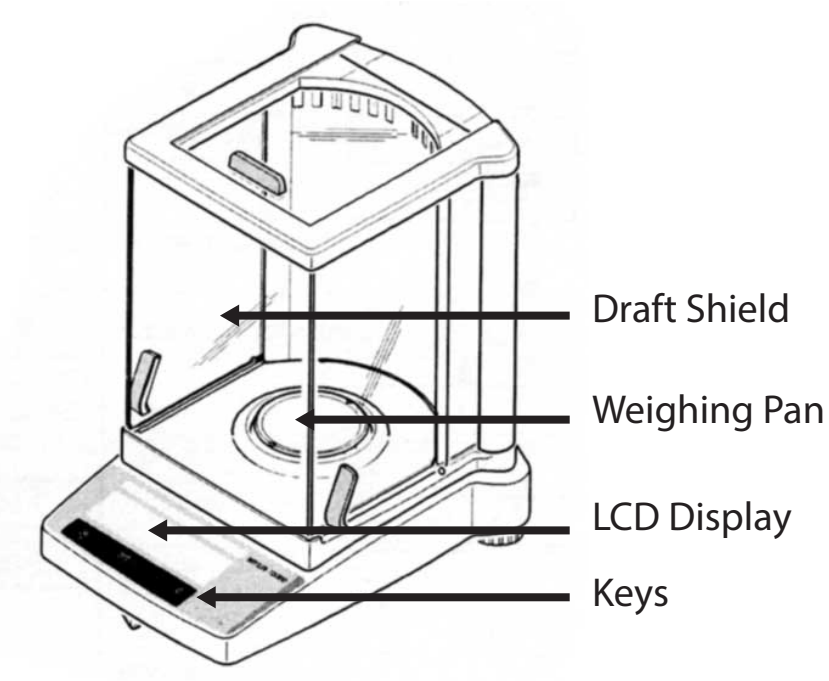


Figure 5. The Analytical Balance

Notice

- Always use weighing boats when weighing solids to protect the balance. To do this, place the plastic weighing boat on the balance pan and be sure it is not touching the sides.
- Always use the balance with extreme care, as it is very expensive.

3. Taring

To measure the mass of sample inside a container, perform the following procedures:

a. Place the empty container (e.g. a weighing boat) on the balance.
b. Press the **0/T** key briefly. The display should read 0.000 g.
c. Add the sample to the container. Read the displayed mass to 0.0001 g.

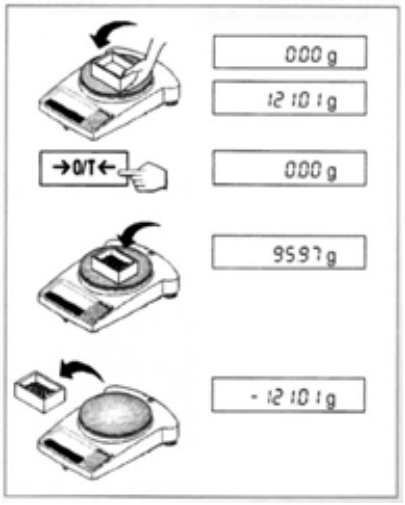


Figure 6. Taring

4. Weighing by Difference

To measure the mass of a sample by difference:

a. Clear the weighing pan. Press **0/T**. The reading should be 0.000 g.
b. Place the container with the sample on the balance. Record the mass.
c. Remove a portion of the sample from the container.
d. The difference between the two readings is the mass of the removed portion of the sample.

Using the Centrifuge

A centrifuge machine is used to separate the different constituents of a solution by their density. In many experiments, you will be required to separate precipitation products from solution using the centrifuge machine.

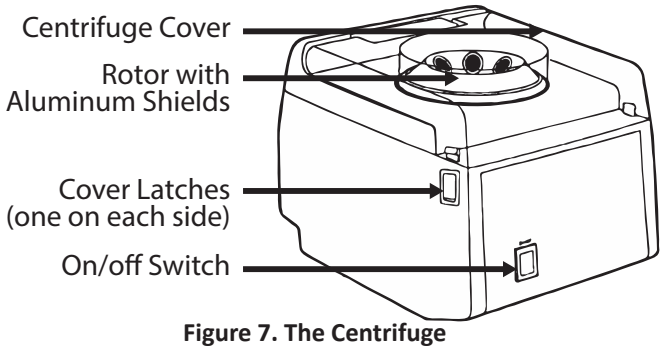


Figure 7. The Centrifuge

1. Procedure

a. Always load centrifuge tubes of about equal weight. Fill another centrifuge tube with water to equal weight to balance.

b. To balance the tubes, place a small beaker on the "quick" balance in your lab room. Weigh your sample tube. Fill another centrifuge tube with water to equal weight (to the nearest 1g).

c. Place the centrifuge tubes in the aluminum shields on opposite sides. The centrifuge tubes should fit inside the aluminum shield snugly. Use a different tube if more than 1/8 inch of the glass is exposed.

d. Close the cover. Lock both sides securely into the latches.

e. Press the On/Off switch to turn on the unit. Press the switch again to turn it off.

Warning

- Improper use of the centrifuge machine may result in serious injury. Follow all safety precautions when operating the centrifuge machine.

2. Safety Precautions

a. Operate the centrifuge only when the cover is securely closed.

b. Never open the cover when the centrifuge is running.

c. Always balance the tubes before loading. Only spin 2, 4, or 6 tubes.

d. **Never spin 1, 3, or 5 tubes.**

e. Turn off the machine immediately if there are signs that the load is unbalanced.

f. Never open the cover before the rotor comes to a **complete** stop.

g. Never stop the rotor with your hand. Serious injury may result.

Using the Hot Plate

The hot plate is used to heat solutions in nearly all experiments performed in the Chemistry 2 laboratory. However, improper use of the hot plate may result in serious injury. Follow all instructions and exercise caution when using the hot plate.

There are a variety of hot plates used in the Chemistry 2 labs, but they all have the same essential features.

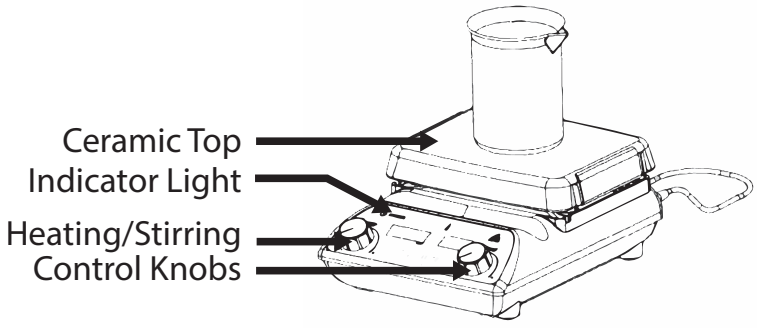


Figure 8. The Hot Plate/Stirrer

1. Features

a. **The Ceramic Top:** The heating surface. The temperature may reach a maximum of over 400 °C. Do not touch the ceramic top. It may cause serious burns. The ceramic top is also very delicate. Clean up spills immediately and avoid hitting the surface with heavy objects.

b. There are four common indicating lights on all models used in the Chemistry 2 laboratories. They are: the Power Indicator, the Heat Indicator, the Stir Indicator, and the Hot Top indicator.

- **Power Indicator:** On if the unit is plugged in to a power source. Check power cord connection if not on.
- **Heat Indicator:** On if the heat is turned on.
- **Stir Indicator:** On if the magnetic stirrer is turned on.
- **Hot Top Indicator:** On if the top has a temperature of over 60°C. Do not unplug the unit if the top plate is still hot.

Warning

- The hot plate may cause serious burns. Avoid touching the top plate and follow all safety precautions.

2. Safety Precautions

a. Keep the power cord away from the heating surface. The cord may melt and cause an electrical hazard.

b. Do not hit the top with heavy objects. It may break if impacted.

c. Do not heat volatile or flammable materials.

d. Do not operate near volatile or flammable materials.

The hot plate must not be used during these experiments:

- **2B.** Colligative Properties
- **2B.** Determination of Avogadro's Number

e. Avoid spilling liquids on the ceramic top. Do not over boil solutions.

It takes approximately 15 minutes to boil 400 mL of water at Heat setting 6. Avoid turning the heat setting too high. Spills from over-boiling will damage the hot plate and may result in personal injury.

f. Never use a container larger than the top plate.

g. Never boil a solution to dryness.

Heating with a Bunsen Burner

In using a Bunsen burner, always use a tight blue flame as shown in the illustration below. Always estimate the appropriate height for the iron support ring before turning on the Bunsen burner. Control the heat transfer by adjusting the distance from the burner to the object. Note that the distances suggested in the manual are measured from the hottest part of the flame to the object.

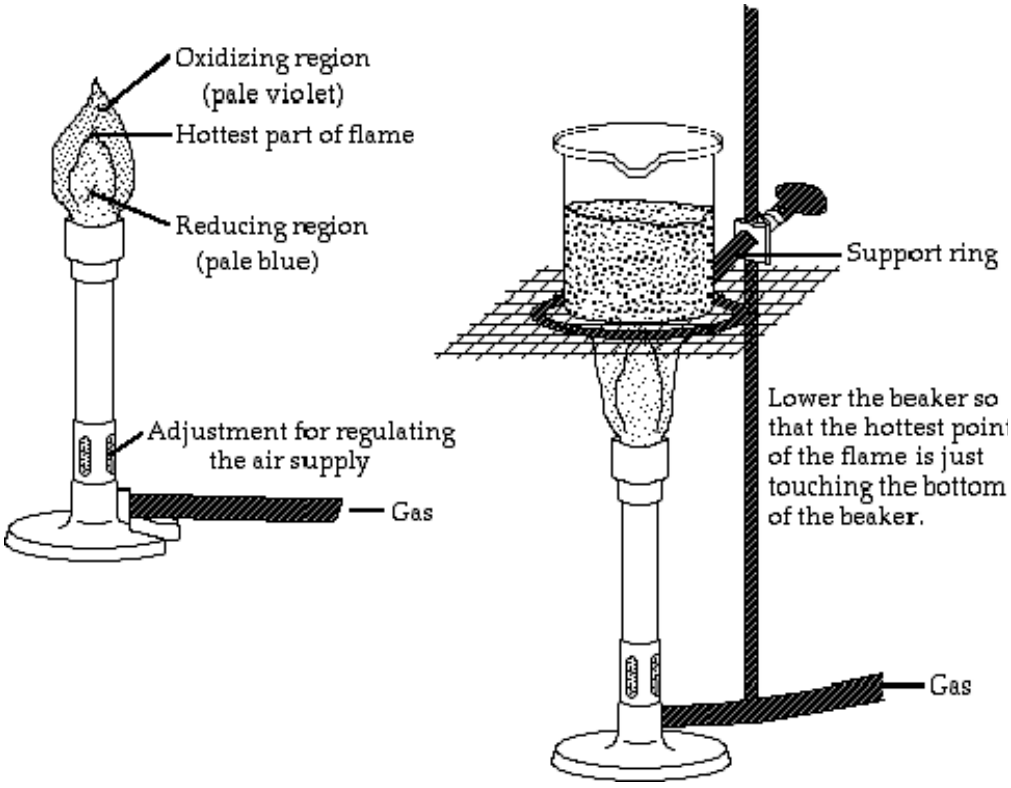


Figure 9. The Bunsen Burner

Warning

- Only use the Bunsen burner when specifically instructed by the lab manual.
- Keep all flammable materials away from the Bunsen burner.
- Heated lab ware including iron rings can be extremely hot and may cause serious burns!

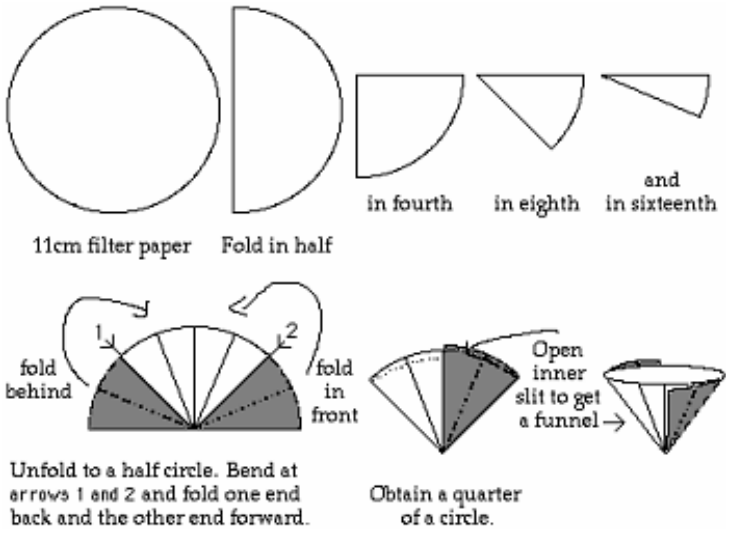
Filtration

You will often need to separate a liquid from a solid. At times you will simply decant, that is, you will carefully pour out the liquid, leaving the solid behind. At other times you will need to filter the solution. To do this you will use filter paper and a funnel. You must first fold the paper in order to accelerate the process; this is shown in Figure 7.

You will then set the paper in the funnel using your wash bottle. To do this simply place the paper into the funnel and add a small amount of water to the bottom of the filter.

Slowly add water to the sides with a circular motion to avoid air bubbles between the paper and the funnel. Once the paper has set, transfer the solution to be filtered. If the solid has settled, decant the liquid through the filter first in order to save time.

Never overwhelm the filter; don't add the solution too quickly and never come to within one centimeter of the top of the paper. Transfer the solid using a wash bottle and rubber policeman, and then wash the solid as directed by the experimental procedure.



Setting up a Titration Apparatus

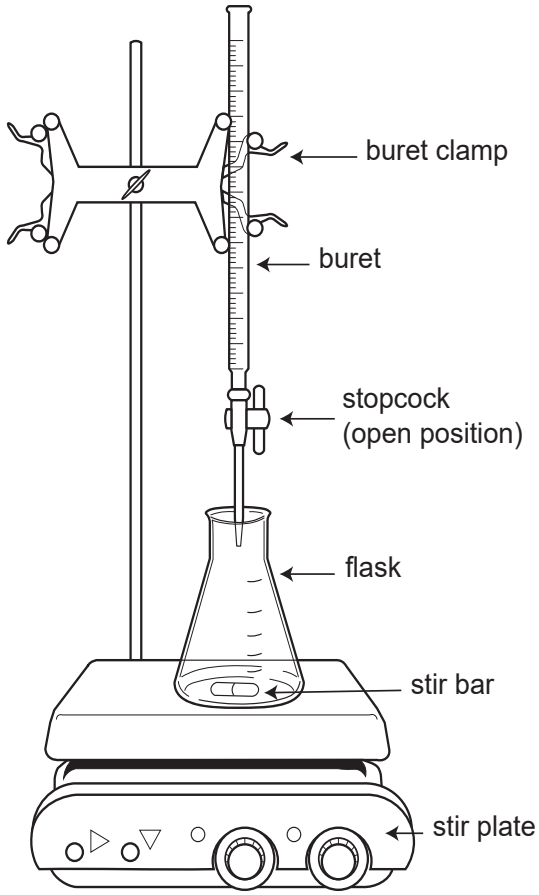


Figure 10. Titration Setup

Titrations often involve the use of strong acids and bases, and properly setting up your titration apparatus can reduce the risk of spills or accidents. Reference Figure 10 and the instructions below to properly set up your titration apparatus.

1. Obtain a stir plate, buret, stir bar, and titration flask. Place the stir plate below the buret clamp located at your lab bench. If applicable, make sure the heating element is turned off.

2. After conditioning and filling the buret, place it securely in the buret clamp. Make sure there is enough room between the buret and stir plate to place the titration flask. Adjust the stir plate so that it is centered underneath the tip of the buret.

3. Use a laboratory tissue to wipe down the tip of the buret. Make one quick stroke downward beginning at the closed stopcock and ending in the air beyond the buret tip. Dispose of the tissue as it may now be contaminated.

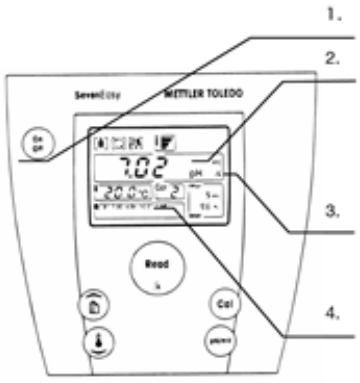
4. Add your stir bar, solution, and indicator to the titration flask, and place the flask underneath the tip of the buret. Turn on the stirrer and slowly increase the stirring speed.

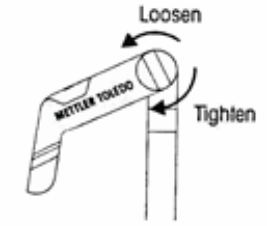
5. Lower the buret tip into flask without touching the flask sides. You are now ready to titrate!

pH Meter Operating Instructions

1. Preparing the pH meter

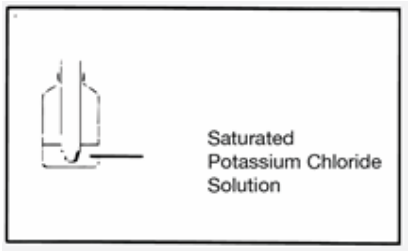
1. Turn on the pH meter.
2. Meter must be in pH mode. If in mV mode, press the **pH/mV** button.
3. Make sure pH meter is showing /Ā. If not shown, press and hold **Read** button for 2 seconds.
4. Lower left window must show **B1 7.00 4.00 10.01 1.68**. If not, ask your TA to adjust the setting.
5. You may adjust the electrode stand to secure the electrode. Loosen the tension knob to adjust arm position and tighten the tension knob before use.





Caution: Do **NOT** place test tubes on electrode stand!

6. Do NOT let electrode dry out. **Always store electrode in saturated KCl solution when not in use.**

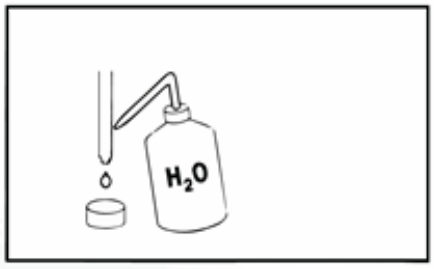


2. Calibrating the pH meter

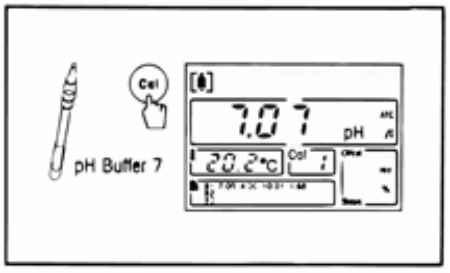
Note: You only need to calibrate the pH meter **once** per lab period.

1. Rinse the electrode with DI water.
2. Blot dry with Kimwipe.

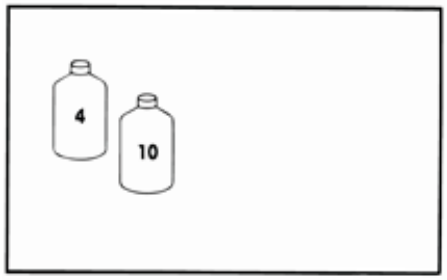
WARNING: Do **NOT** rub the electrode with Kimwipe. Rubbing the electrode may build up static charge and damage the electrode.



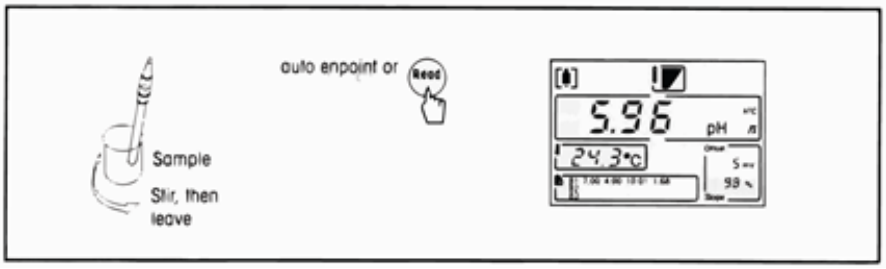
3. Place electrode in pH 7 buffer standard (yellow).
4. Press the **Cal** button.
5. Wait for the display to stop blinking.



6. Repeat step B-1 to B-5 with the pH 4 buffer standard (red) and then with the pH 10 buffer standard (blue).



3. Measure the pH of sample

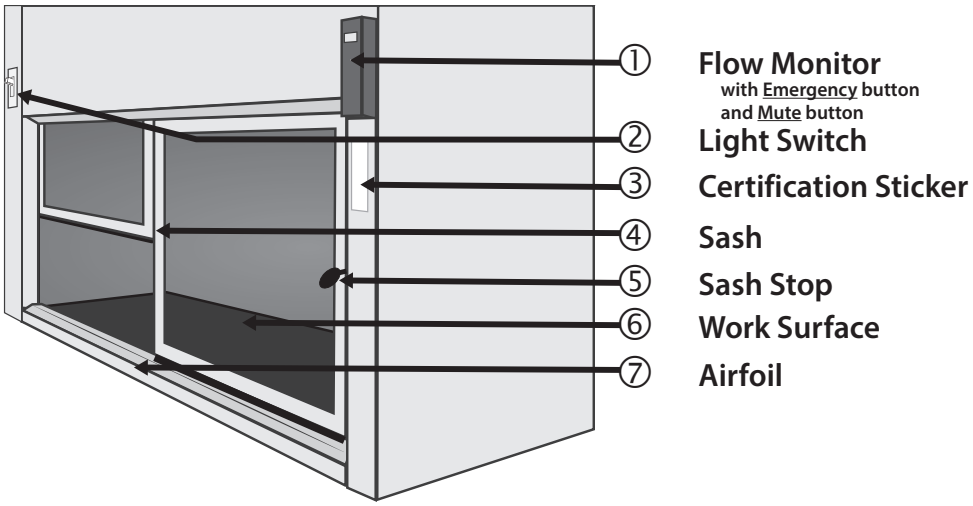


1. After calibration, place the electrode in sample solution and press **Read**.
2. Wait for the reading to stabilize.

Fume Hood Use and Safety

The fume hoods in the laboratory protect personnel from hazardous materials and inhalation of toxic materials.

1. Features of the Fume Hood



2. Before using the fume hood

1. Check the **certification sticker** (③). The Fume Hood is tested and certified every year.
2. Check the flow monitor (①).

   Laboratory fume hood should have 100 ft/min face velocity or more. Lower the sash if to increase airflow. If airflow does not reach 100 ft/min, stop work in the fume hood and contact safety personnel immediately.
3. Turn on light switch (②).

3. Guidelines for working with the fume hood

1. Lift the sash up slowly about 12 inches. Never raise the sash above the sash stop (⑤).
2. Always place lab equipment at least six inches away from the edge and inside the fume hood as much as possible.
3. Do not rest body parts on the edge or the Airfoil (⑦).
4. Do not place glassware or chemicals on the Airfoil (⑦).
5. Move unused equipment and chemicals away. Remove your glassware when done.

Remember, you are sharing the fume hood with 23 other students. Remove your glassware as soon as possible and clean your glassware. Do **NOT** abandon your lab ware in the fume hood!

6. When increased airflow is needed, press the **Emergency** button and the **Mute** button.
7. **Clean up spills immediately.**
8. **Cap all containers immediately.**
9. Turn off **Emergency** mode and close hood sash all the way at the end of lab.

4. Using the fume hoods in the Chemistry 2 Laboratories

1. **Always use the fume hood when directed by the Laboratory Manual.**

Certain reactions in the Chemistry 2 curriculum generate toxic or flammable gases. Follow instructions to protect yourself and others in the lab.

2. Many hazardous chemicals are kept in the fume hood. Never remove these containers unless specifically directed by the Laboratory Manual.
3. All Hazardous Waste containers for the Chemistry 2 course are kept in the fume hood.

5. Fume Hood Emissions

1. Minimize fume hood emissions to protect the environment and air quality.
2. Never evaporate waste in the fume hood.
3. Minimize use of volatile liquids. Close and seal after using.

If you have questions, contact your TA or safety coordinator.

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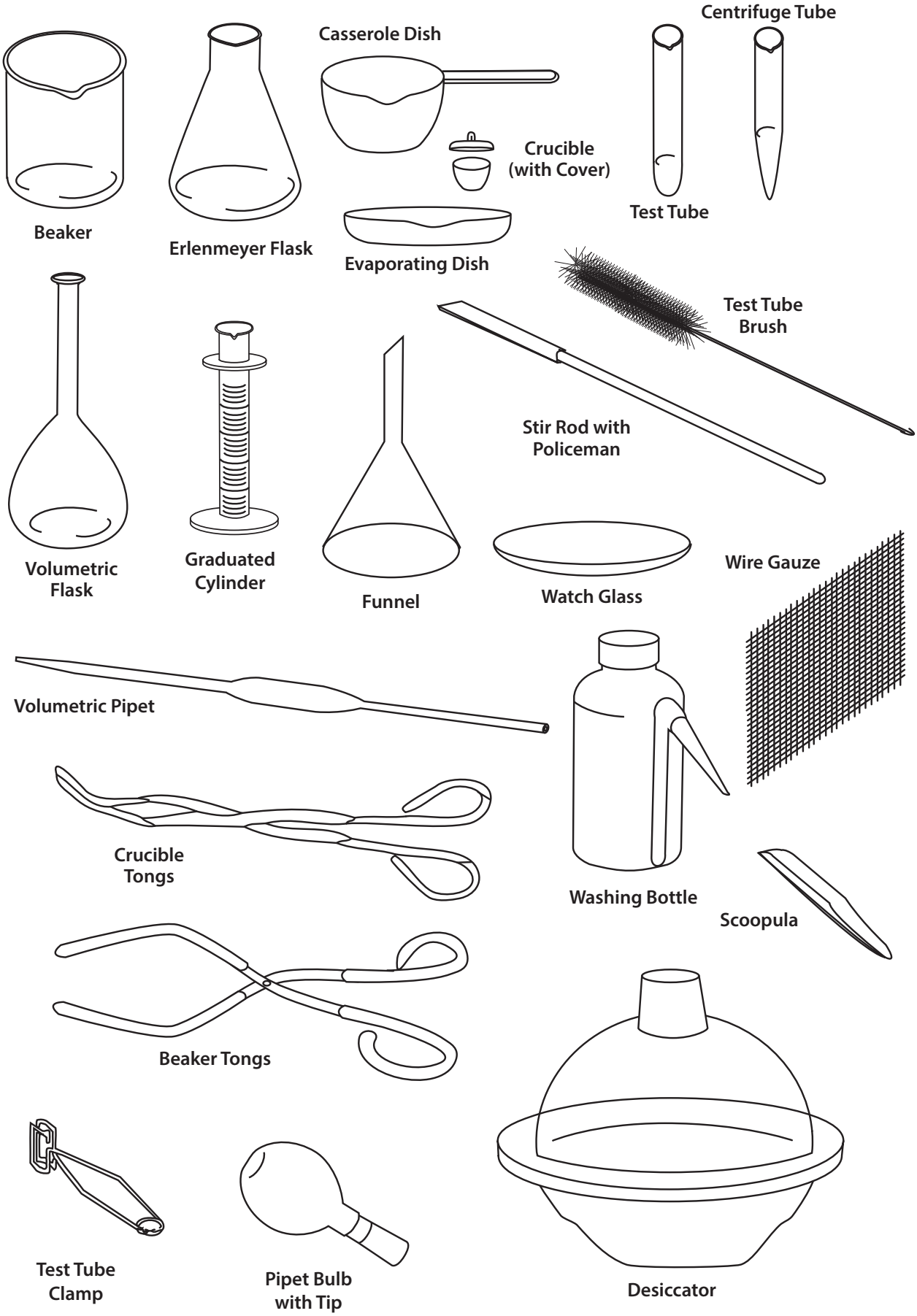
Beaker
Erlenmeyer Flask
Casserole Dish
Crucible
(with Cover)
Evaporating Dish
Centrifuge Tube
Test Tube
Test Tube
Brush
Stir Rod with
Policeman
Volumetric
Flask
Graduated
Cylinder
Funnel
Watch Glass
Wire Gauze
Volumetric Pipet
Crucible
Tongs
Washing Bottle
Scoopula
Beaker Tongs
Test Tube
Clamp
Pipet Bulb
with Tip
Desiccator

Locker Inventory

Procedure for beginning of quarter locker check-in:

1. Count the numbers of items currently present in locker.
2. Place excess items from locker into the extra glassware box in the back of lab.
3. Return community supplies to the appropriate storage location.
4. Check out missing items from the following sources:
   a) from the extra glassware box in the back of lab
   b) from the Dispensary service window (1st floor, Esau Hall 1060E)
5. Clean and dry all equipment.

CHEMISTRY 2 LOCKER LIST

Glassware			Porcelain		
# present	**# total**	**Description**	**# present**	**# total**	**Description**
	1	100 mL Beaker		1	150 mL Casserole Dish
	1	150 mL Beaker		1	Evaporating Dish
	1	250 mL Beaker		2	Crucible
	1	400 mL Beaker		2	Crucible Cover
	1	800 mL Beaker	**Plastic Ware**		
	2	50 mL Erlenmeyer Flask	**# present**	**# total**	**Description**
	2	125 mL Erlenmeyer Flask		1	250 mL Washing Bottle
	2	250 or 500 mL Erlenmeyer Flask		1	Short Stem Funnel
	1	100 mm Watch Glass		2	1 L Bottle, square
	2	Glass Stir Rod		1	Desiccator
	10	Test Tubes (rounded end)		1	Plastic Test Tube Rack
	6	Centrifuge Tubes (pointed end)	**Other**		
	2	Thermometer, non-mercury	**# present**	**# total**	**Description**
	2	25 mL Volumetric Flask		1	Centrifuge Tube Brush (pointed)
	1	250 mL Volumetric Flask		1	Test Tube Brush (round)
	1	10 mL Graduated Cylinder		1	Vial, Alkacid Test Paper
	1	25 mL Graduated Cylinder		1	Sponge
Metal Equipment				2	Rubber Policeman
# present	**# total**	**Description**			
	1	Wire Gauze Square			
	1	Scoopula			

COMMUNITY SUPPLIES (not in student lockers)

Lab Island Lockers	Wall Side Drawers
8″ Extension Clamp	Beaker Tongs
Clamp Holder	Crucible Tongs
4″ Support Ring	Test Tube Clamp
Overhead Storage Cabinet	Bunsen Burner
Pipet Bulb	Silicone Rubber Tubing
1 mL Pipet	**Storage Cabinet**
5 mL Pipet	25 mL Buret
10 mL Pipet	

Procedure for end of quarter locker check-out:

1. Clean and dry all equipment.
2. Count the numbers of items currently present in locker.
3. Place excess items from locker into the extra glassware box in the back of lab.
4. Return community supplies to the appropriate storage location.
5. Check out missing items from the following sources:
   a) from the extra glassware box in the back of lab
   b) from the Dispensary service window (1st floor, Esau Hall 1060E)

CHEMISTRY 2 LOCKER LIST

Glassware			Porcelain		
# present	# total	Description	# present	# total	Description
	1	100 mL Beaker		1	150 mL Casserole Dish
	1	150 mL Beaker		1	Evaporating Dish
	1	250 mL Beaker		2	Crucible
	1	400 mL Beaker		2	Crucible Cover
	1	800 mL Beaker	**Plastic Ware**		
	2	50 mL Erlenmeyer Flask	# present	# total	Description
	2	125 mL Erlenmeyer Flask		1	250 mL Washing Bottle
	2	250 or 500 mL Erlenmeyer Flask		1	Short Stem Funnel
	1	100 mm Watch Glass		2	1 L Bottle, square
	2	Glass Stir Rod		1	Desiccator
	10	Test Tubes (rounded end)		1	Plastic Test Tube Rack
	6	Centrifuge Tubes (pointed end)	**Other**		
	2	Thermometer, non-mercury	# present	# total	Description
	2	25 mL Volumetric Flask		1	Centrifuge Tube Brush (pointed)
	1	250 mL Volumetric Flask		1	Test Tube Brush (round)
	1	10 mL Graduated Cylinder		1	Vial, Alkacid Test Paper
	1	25 mL Graduated Cylinder		1	Sponge
Metal Equipment				2	Rubber Policeman
# present	# total	Description			
	1	Wire Gauze Square			
	1	Scoopula			

COMMUNITY SUPPLIES (not in student lockers)

Lab Island Lockers	Wall Side Drawers
8" Extension Clamp	Beaker Tongs
Clamp Holder	Crucible Tongs
4" Support Ring	Test Tube Clamp
Overhead Storage Cabinet	Bunsen Burner
Pipet Bulb	Silicone Rubber Tubing
1 mL Pipet	**Storage Cabinet**
5 mL Pipet	25 mL Buret
10 mL Pipet	

Periodic Table of the Elements

1 IA 1A	2 IIA 2A	3 IIIB 3B	4 IVB 4B	5 VB 5B	6 VIB 6B	7 VIIB 7B	8	9 VIII 8	10	11 IB 1B	12 IIB 2B	13 IIIA 3A	14 IVA 4A	15 VA 5A	16 VIA 6A	17 VIIA 7A	18 VIIIA 8A
1 **H** Hydrogen 1.008																	2 **He** Helium 4.003
3 **Li** Lithium 6.941	4 **Be** Beryllium 9.012											5 **B** Boron 10.811	6 **C** Carbon 12.011	7 **N** Nitrogen 14.007	8 **O** Oxygen 15.999	9 **F** Fluorine 18.998	10 **Ne** Neon 20.180
11 **Na** Sodium 22.990	12 **Mg** Magnesium 24.305											13 **Al** Aluminum 26.982	14 **Si** Silicon 28.086	15 **P** Phosphorus 30.974	16 **S** Sulfur 32.066	17 **Cl** Chlorine 35.453	18 **Ar** Argon 39.948
19 **K** Potassium 39.098	20 **Ca** Calcium 40.078	21 **Sc** Scandium 44.956	22 **Ti** Titanium 47.867	23 **V** Vanadium 50.942	24 **Cr** Chromium 51.996	25 **Mn** Manganese 54.938	26 **Fe** Iron 55.845	27 **Co** Cobalt 58.933	28 **Ni** Nickel 58.693	29 **Cu** Copper 63.546	30 **Zn** Zinc 65.38	31 **Ga** Gallium 69.723	32 **Ge** Germanium 72.631	33 **As** Arsenic 74.922	34 **Se** Selenium 78.971	35 **Br** Bromine 79.904	36 **Kr** Krypton 84.798
37 **Rb** Rubidium 84.468	38 **Sr** Strontium 87.62	39 **Y** Yttrium 88.906	40 **Zr** Zirconium 91.224	41 **Nb** Niobium 92.906	42 **Mo** Molybdenum 95.95	43 **Tc** Technetium 98.907	44 **Ru** Ruthenium 101.07	45 **Rh** Rhodium 102.906	46 **Pd** Palladium 106.42	47 **Ag** Silver 107.868	48 **Cd** Cadmium 112.414	49 **In** Indium 114.818	50 **Sn** Tin 118.711	51 **Sb** Antimony 121.760	52 **Te** Tellurium 127.6	53 **I** Iodine 126.904	54 **Xe** Xenon 131.294
55 **Cs** Cesium 132.905	56 **Ba** Barium 137.328	57-71	72 **Hf** Hafnium 178.49	73 **Ta** Tantalum 180.948	74 **W** Tungsten 183.84	75 **Re** Rhenium 186.207	76 **Os** Osmium 190.23	77 **Ir** Iridium 192.217	78 **Pt** Platinum 195.085	79 **Au** Gold 196.967	80 **Hg** Mercury 200.592	81 **Tl** Thallium 204.383	82 **Pb** Lead 207.2	83 **Bi** Bismuth 208.980	84 **Po** Polonium [208.982]	85 **At** Astatine 209.987	86 **Rn** Radon 222.018
87 **Fr** Francium 223.020	88 **Ra** Radium 226.025	89-103	104 **Rf** Rutherfordium [261]	105 **Db** Dubnium [262]	106 **Sg** Seaborgium [266]	107 **Bh** Bohrium [264]	108 **Hs** Hassium [269]	109 **Mt** Meitnerium [278]	110 **Ds** Darmstadtium [281]	111 **Rg** Roentgenium [280]	112 **Cn** Copernicium [285]	113 **Nh** Nihonium [286]	114 **Fl** Flerovium [289]	115 **Mc** Moscovium [289]	116 **Lv** Livermorium [293]	117 **Ts** Tennessine [294]	118 **Og** Oganesson [294]

Lanthanide Series	57 **La** Lanthanum 138.905	58 **Ce** Cerium 140.116	59 **Pr** Praseodymium 140.908	60 **Nd** Neodymium 144.243	61 **Pm** Promethium 144.913	62 **Sm** Samarium 150.36	63 **Eu** Europium 151.964	64 **Gd** Gadolinium 157.25	65 **Tb** Terbium 158.925	66 **Dy** Dysprosium 162.500	67 **Ho** Holmium 164.930	68 **Er** Erbium 167.259	69 **Tm** Thulium 168.934	70 **Yb** Ytterbium 173.055	71 **Lu** Lutetium 174.967
Actinide Series	89 **Ac** Actinium 227.028	90 **Th** Thorium 232.038	91 **Pa** Protactinium 231.036	92 **U** Uranium 238.029	93 **Np** Neptunium 237.048	94 **Pu** Plutonium 244.064	95 **Am** Americium 243.061	96 **Cm** Curium 247.070	97 **Bk** Berkelium 247.070	98 **Cf** Californium 251.080	99 **Es** Einsteinium [254]	100 **Fm** Fermium 257.095	101 **Md** Mendelevium 258.1	102 **No** Nobelium 259.101	103 **Lr** Lawrencium [262]