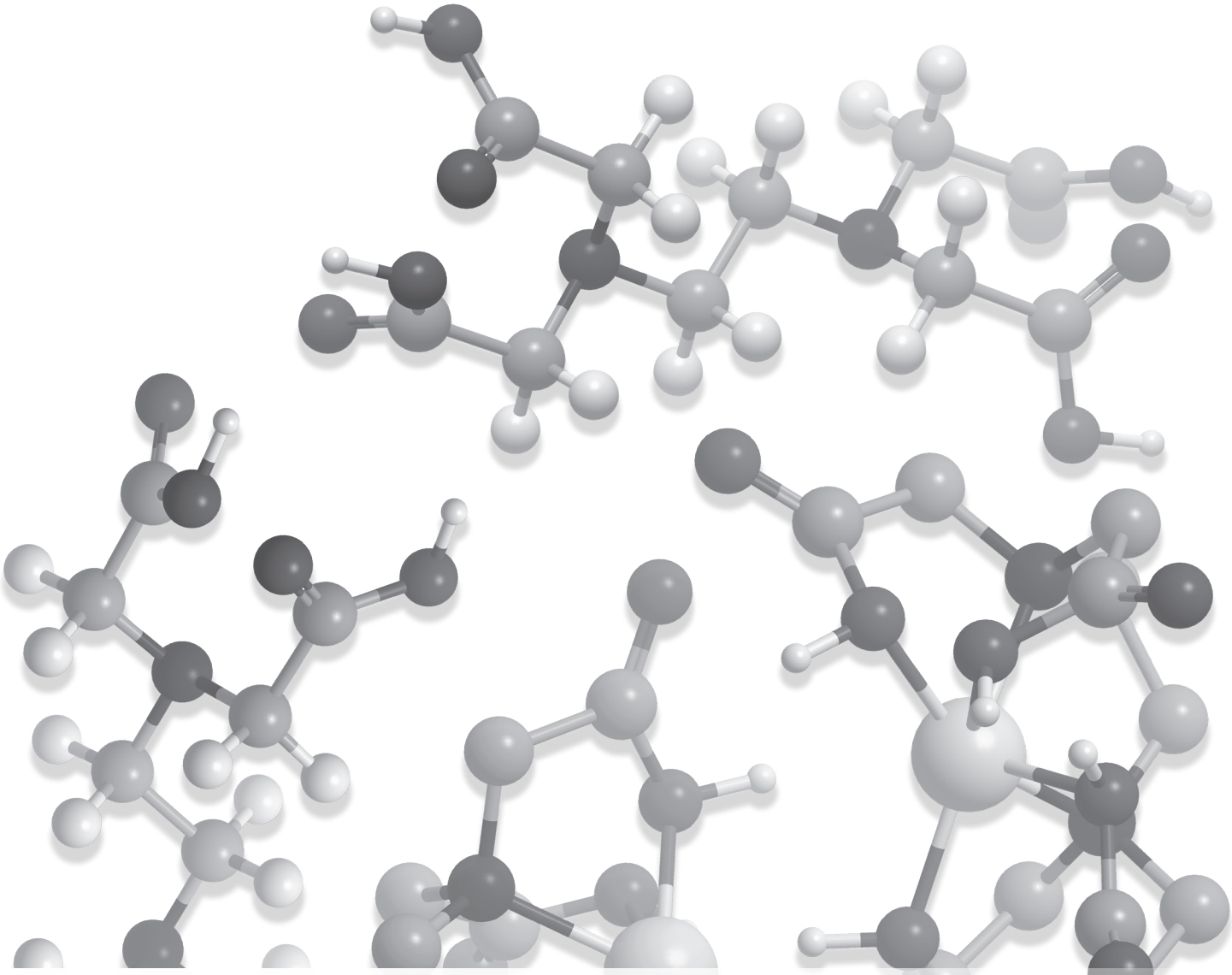


Chemistry 2C Lab Manual

Standard Operating Procedures

Summer Session 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 Sciences Lab Building**

Location of Safety Equipment Nearest to Your Laboratory

Safety Shower _____

Eye Wash Fountain _____

Fire Extinguisher _____

Fire Alarm _____

Safety Chemicals _____

Revision Date: June 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
Redox Titrations	1
Nomenclature Quiz	N/A
Electrochemical Cells	1
EDTA Titrations	1
Qualitative Analysis	2
Synthesis of TM Compounds	1
Spectroscopy	1
Kinetics	1
Vitamin C	1
On-Line Prelab Quizzes (seven)	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 may take the quiz a second time. Because the questions are chosen randomly, different questions may be generated on the second 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

Redox Titrations

Introduction

In this experiment you are going to use a pair of oxidation-reduction reactions to determine the concentration of an unknown bleach. First, you are going to standardize a freshly prepared solution of sodium thiosulfate ($Na_2S_2O_3$). You will do this by reacting iodate (IO_3^-) with excess iodide (I^-) in acidic solution to produce a known quantity of iodine (I) and water. The iodine produced will be titrated with a sodium thiosulfate solution. The products of the reaction between iodine and thiosulfate are the iodide ion and the tetrathionate ion ($S_4O_6^{2-}$). By using a known amount of iodate and stoichiometric calculations you can calculate the exact concentration of the thiosulfate solution you have prepared.

In the second part of the experiment you will use your standardized thiosulfate solution to analyze an unknown bleach. Bleach contains sodium hypochlorite (NaClO) that reacts with iodide to produce iodine, chloride ion, and water. The amount of iodine produced can then be determined by titration with your standard sodium thiosulfate.

As pre-laboratory preparation, you should read the Redox sections in your text. Redox reactions are much more complicated than simple acid/base reactions. You should ***balance all the reactions*** described in this experiment before coming to the laboratory.

What kind of bleach is it?

Bleach is a collective term that refers to a variety of chemical products that are used industrially or domestically to remove color from fabrics, fibers, and other materials. One of the most common type of bleach found in stores and supermarkets is a dlute solution of sodium hypochlorite, better known as "liquid bleach." It is typically used as a stain remover during cleaning and laundry. Bleach can also be effective in killing bacteria and viruses, making it a useful tool for disinfection in homes, hospitals, and other environments.

Learning Goals

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

Laboratory	• Preparing a secondary standard • Titrations • Using volumetric pipets
Conceptual	• Redox reactions • Oxidation states
Data Analysis	• Calculating mass of an unknown amount of solid in solution using primary and secondary standards • Calculating the mass of a compound in a solution of unknown concentration using primary and secondary standard solutions

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
3 M Sulfuric Acid	< 15 mL
1 M Sodium Thiosulfate	According to calculation
Potassium Iodate *(solid)*	According to calculation
1% Starch	6 mL
Potassium Iodide *(solid)*	6 grams
Bleach Unknown	< 2 grams

Safety First

Wear your goggles and gloves throughout this experiment.

Part I. Standardization of Sodium Thiosulfate Solution

Below is a rough outline of the titration procedure: you should elaborate and provide further details such as the amounts used of each reagent in your laboratory report. The outline of the procedure also contains some important information that will assist you in performing an effective titration.

Important Tips

- The chemicals listed above will be provided for you in the lab. Do not contaminate the chemicals.
- The solutions in the reagent bottles should be colorless. If they are brown, they have been contaminated and should be returned to the dispensary.
- The most common causes of contamination are leaving the caps off the chemical bottles and using the same disposable pipet for more than one chemical.
- Be vigilant! Do not be the person in your lab room that causes these problems!

1. Preparation of the sodium thiosulfate secondary standard solution.

You should dilute the sodium thiosulfate solution to approximately 0.05 M. This will give you reasonable volumes for your titrations. Plan on making about 200 mL of the sodium thiosulfate solution.

2. Preparation of the potassium iodate standard solution.

Prepare a 250 mL solution of approximately 0.01 M potassium iodate in a 250 mL volumetric flask and share the solution between 2–3 groups. Be sure to use volumetric glassware to transfer this iodate solution into your titration flask.

Calculate the final molarity of the potassium iodate to **three significant figures**.

This method will speed up your titrations in two ways. You will spend far less time at the balance, and potassium iodate takes a few minutes to dissolve so you only have to wait once instead of several times.

3. Preparation of the iodine solution standard.

Since you know very precise information about your iodate solution, make iodate your limiting reagent. If you do your reactions with **10 mL of iodate solution**, you will need about one gram of potassium iodide and 1 mL of the 3 M sulfuric acid.

Use your 10.00 mL volumetric pipet to measure 10.00 mL of the potassium iodate. This mixture will turn a dark red or brown (either color is acceptable).

In an acidic solution, iodate and iodide react to produce iodine. Sulfuric acid is chosen for this reaction since the sulfate anion will not interfere with this particular set of redox reactions.

Save time by carefully measuring only your limiting reagent. The excess reagents can be measured out much more generally. On the other hand, do not waste chemicals by adding large excesses of the other reagents.

Add 30.0 mL of deionized water to the titration flask so that you can easily see the color changes.

Tips on filling a buret

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

4. Titration of the iodine solution with the sodium thiosulfate.

Titrate the iodine solution with the sodium thiosulfate solution you have made. Do not add the starch indicator until the titration is almost finished.

As you perform the titration, the solution color should go from brown to yellow. Once the solution fades to yellow you should add 1 mL of the starch indicator. This sharpens the endpoint.

In concentrated iodine solutions, the starch complex can tie up some of the dissolved iodine molecules, effectively removing them from the titration and affecting the accuracy of the results.

Clean-up

- Add **0.5 grams of sodium bicarbonate** to the titrated flask before disposing down the drain with copious amount of water.

Do at least three trials. Perform the titration immediately upon mixing the chemicals. Though it is tempting to use "assembly line" techniques and prepare all of your flasks at once and then titrate them all, this can also adversely affect your results. The iodine in the solution can clump together over time and become more and more difficult to dissolve.

Report your average molarity of $Na_2S_2O_3$ standardized solution, and standard deviation. The post-lab exercises will guide you through these calculations.

Part II. Analysis of your Bleach Unknown

Once you have standardized your sodium thiosulfate solution, you can move on to the analysis of your bleach unknown. Plan a titration procedure that will allow you to determine the mass percentage of sodium hypochlorite in a bleach sample.

Your procedure should be fundamentally the same as for the standardization, starting at Part I Step 3, but bleach (hypochlorite) will take the place of iodate in the reactions. As in Part I, combine each of your bleach samples with about 1 g of potassium iodide, 1 mL of 3 M sulfuric acid, and 30.0 mL of deionized water. The tips for the standardization procedure also apply for your unknown analysis.

Plan to use 0.5 grams of bleach unknown in each of your titrations. Remember to either record the weight of or tare the empty titration flask before you measure the mass of the bleach. Record the mass of bleach used for each trial to a thousandth of a gram.

Like in Part I, add about 1 gram of potassium iodide and 1 mL of 3 M sulfuric acid to your titration flask. Again, add 30.0 mL of deionized water to the titration flask so that you can easily see color changes.

Be sure to write down your unknown number, because you will need it handy for the post-lab analysis.

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.

Clean-up

- Neutralize any acidic solutions with **0.5 g sodium bicarbonate** before disposal.
- Rinse the buret and all glassware with deionized water.
- All non-corrosive solutions can be disposed down the drain with copious amounts of water.
- If time permits, now would be a good time to clean any other dirty glassware in your locker. Be sure that all glassware is returned to the proper place and that your laboratory bench has been wiped down with water.

Data Analysis

Part I. Standardization of Sodium Thiosulfate Solution

The following series of questions pertains to Part I of the Redox Titration Experiment, where you are to calculate the molarity of the sodium thiosulfate standard solution.

1. What is the mass in **GRAMS** of the potassium iodate you used to prepare your primary standard solution? Your mass precision should be reported to a thousandth of a gram.

2. If one had weighed out precisely 0.500 g of KIO_3 for the primary standard solution and dissolved it in enough deionized water to make a 250 mL solution, the molarity of that solution would be 0.00935 M. Using the mass of potassium iodate (reported to a thousandth of a gram) you used to prepare the primary standard solution, calculate the molarity of your primary standard KIO_3 solution.

3. For each of your trials, what is the precise volume in mL of the potassium iodate solution used in the standardization of sodium thiosulfate (e.g. 10.00 mL)?

4. For each of your trials, what is the precise volume in mL of the sodium thiosulfate solution used in the titration of your potassium iodate solution (e.g. 11.43 mL)?

5. For each of your trials, calculate the molarity of your sodium thiosulfate solution using the molarity of your potassium iodate solution, the volume of potassium iodate solution used, and the volume of sodium thiosulfate used in the titration of your potassium iodate solution.

6. Calculate the average molarity of your sodium thiosulfate solution.

7. Calculate the standard deviation of the molarities of the sodium thiosulfate solution.

Part II. Analysis of Bleach

The following series of questions pertains to Part II of the Redox Titration Experiment, where you are to calculate the mass percent of sodium hypochlorite in your sample of bleach.

8. For the reaction of hypochlorite anion with iodide anion, the iodide anion acts as the reducing agent according to the oxidation half-reaction, $2I^-_{(aq)} \rightarrow I_{2(aq)} + 2e^-$. What is the reduction half-reaction for the reaction of hypochlorite anion with iodide anion?

9. For the reaction of thiosulfate anion with iodine, the thiosulfate anion acts as the oxidizing agent according to the oxidation half-reaction, $2S_2O_3^{2-}{}_{(aq)} \rightarrow S_4O_6^{2-}{}_{(aq)} + 2e^-$. What is the reduction half-reaction for the reaction of thiosulfate anion with iodine?

10. Notice that the effective analytical connection for analysis of hypochlorite anion by the thiosulfate anion in this experiment is the sum of the two reactions: 1) hypochlorite anion with iodide anion to form iodine and 2) thiosulfate anion with the iodine formed in reaction one. What is the stoichiometric factor that relates moles of thiosulfate anion needed to react with each mole of hypochlorite anion in the bleach sample?

11. For each of your trials, what is the mass of your bleach sample in **GRAMS**? Your mass precision should be reported to a thousandth of a gram

12. For each of your trials, what is the precise volume in **milliliters** of sodium thiosulfate solution used in the titration of your bleach sample (e.g. 11.81 mL)?

13. Using the volumes of sodium thiosulfate solution, the mass of bleach sample, and the average molarity of the sodium thiosulfate solution, calculate the mass percent of NaClO for each bleach sample.

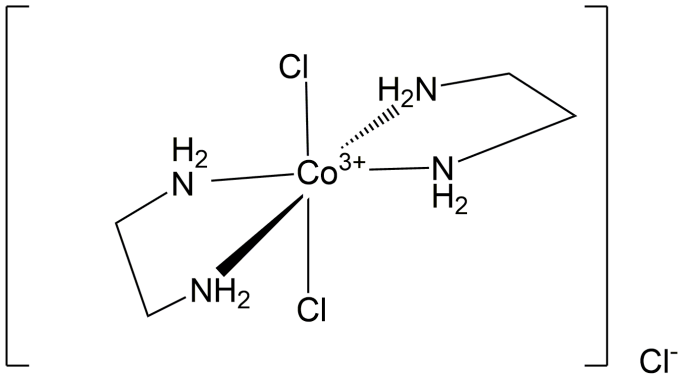
14. Calculate the average mass percent of NaClO in your bleach sample.

15. Calculate the standard deviation of the mass percent of NaClO in your bleach samples.

16. Calculate the 90% confidence limit for the average mass percent of the NaClO in your bleach sample.

17. Record your unknown number.

Online Test: Nomenclature of Transition Metal Complexes



Cobalt Complex

One of the most interesting aspects of transition metal chemistry is the formation of coordination compounds, which are often referred to as transition metal complexes. You will be studying some of these transition metal complexes in Chemistry 2C. Because of their unique structure, transition metals have their own system of nomenclature.

See your course syllabus, TA, or your course instructor for the due date of the Nomenclature quiz.

Electrochemical Cells

Introduction

The use of electrochemical cells to convert the Gibbs energy stored in the constituent half-reactions into electrical work is of enormous industrial as well as fundamental significance. We have all used batteries and these are simply galvanic cells similar to those you will construct in this experiment. In the laboratory, a typical electrochemical (or galvanic) cell has the following general construction:

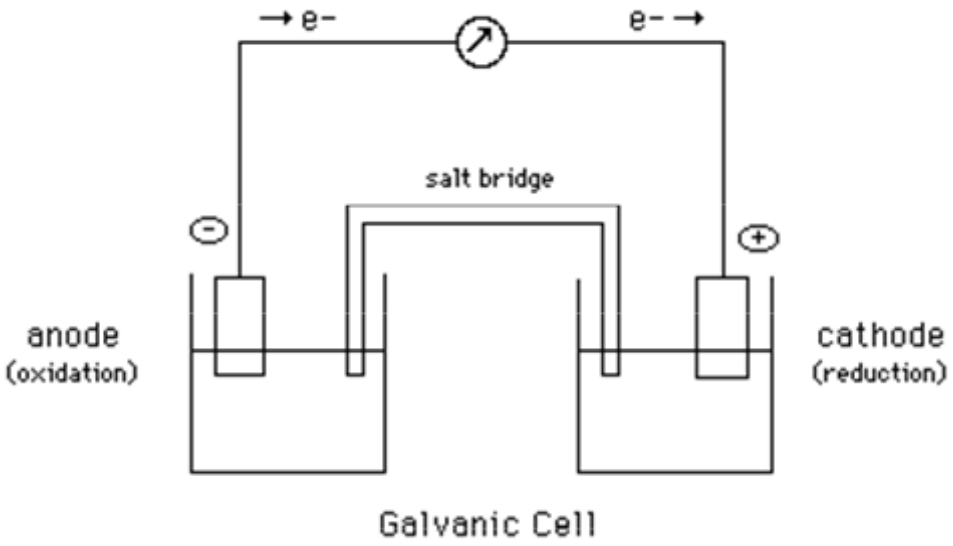


Figure 1: A Schematic of a Galvanic Cell

In Figure 1, there are two electrode-compartments, each of which contains an electrode and the constituents of the half-reaction. In many instances, the electrode is actually one of the chemical components of the half-reaction. For example, the copper electrode is involved directly in the half reaction, $Cu^{2+} + 2e^- \rightarrow Cu$. In other cases, however, the electrode does not participate in the chemistry of the half-reaction, but merely provides an inert conducting surface on which the electron exchange occurs. For example, when the half reaction $Fe^{3+} + e^- \rightarrow Fe^{2+}$ is studied, a Pt or C electrode rather than an iron electrode is used. This is because on an iron wire, both $Fe^{3+} + 3e^- \rightarrow Fe_{(s)}$ and $Fe^{2+} + 2e^- \rightarrow Fe_{(s)}$ could occur, rather than the Fe^{3+} / Fe^{2+} reaction that is of interest.

The two electrode compartment must be separated by a barrier that permits ions to migrate inside the cell, but does not allow the contents of the chambers to mix. A glass tube that is filled with a gel saturated with a strong electrolyte such as KNO_3 is often used. In this case, the K^+ and NO_3^- ions create a "salt bridge" for the electrochemical cell.

Electrochemical cells have both a magnitude for the measured voltage and a polarity. An electrode that is positive, relatively speaking, must be deficient in electrons, and hence a reduction must be taking place at that electrode. Conversely, an electrode that appears negative has a surplus of electrons. Hence, electrons are being released into it by an oxidative half-reaction. By definition, oxidation occurs at an anode. Hence, the (-) pole of an electrochemical cell is necessarily its anode, and the (+) pole is its cathode (where the reduction occurs).

The directions for this experiment exploit the fact that electrochemical cells can be described very efficiently by using conventional "cell diagrams." A possible diagram for a galvanic cell that employs the net ionic reaction,

$$2Ag^{+}(aq) + Cu(s) \rightarrow Cu^{2+}(aq) + 2Ag(s)$$

is as follows:

$$Cu(s) \mid Cu(NO_3)_2(aq)\ (0.1\ M) \parallel AgNO_3(aq)\ (0.1\ M) \mid Ag(s)$$

The corresponding measured conventional cell voltage is roughly +0.4 V.

The cell diagram contains the information necessary to construct the electrochemical cell. The cell diagram has a "right hand" electrode (here a piece of silver wire) and a "left hand" electrode (here a piece of copper wire). A single vertical line means that the two species flanking it have different phases. In this case, the two phases are the solid wires and the liquid solutions whose concentrations are specified inside the parentheses. The double vertical line implies some type of chemically inert salt bridge connecting the two compartments in question.

The algebraic sign of a cell containing a spontaneous reaction is always a positive voltage. A negative voltage reading is possible in this experiment even though all the reactions are spontaneous. If a negative voltage is measured, then the cell diagram is written in reverse of the spontaneous direction of the cell. A correct cell diagram of a spontaneous redox reaction has the anode written on the left of the double vertical line and the cathode written to the right.

In this experiment you will construct and measure the voltage of electrochemical cells that involve the half-reactions (in alphabetical order): $Cu^{2+} + 2e^{-} \rightarrow Cu$, $Fe^{3+} + e^{-} \rightarrow Fe^{2+}$, $Pb^{2+} + 2e^{-} \rightarrow Pb$, $Zn^{2+} + 2e^{-} \rightarrow Zn$. From your results you will be able to determine the relative positions of these half-reactions in a Table of Standard Potentials. You will also confirm that the potentials of half-reactions are concentration dependent. As you know, in order to calculate the voltages of non-standard cells (not 1 M and 1 atm), you need to use the Nernst equation.

A useful form of the Nernst equation is in your text in the Electrochemistry section. Review the appropriate chapter to help you prepare for this experiment.

This experiment is quite easy and short in terms of the actual collection of data. However, the write-up is difficult and requires a good understanding of electrochemistry. Students in the past have found it very helpful to take the time to do each step slowly and try to understand what is happening. They also said that it is critical to begin the write-up while in the laboratory in order to repeat steps that have become confusing or to get assistance from the TA or fellow students. You are strongly encouraged to follow this advice!

Electrochemistry in Today's World

Electrochemistry plays an essential role in various apects of contemporary life , such as constructing roads and bridges that can withstand corrosion, monitoring the quality of potable water, producing hydrogen without carbon emission, and developing biosensors for diagnostic purposes. it serves as the fundamental science behind batteries, solar cells, fuel cells, and the conversion of CO_2 into valuable products. As an important pillar in society's transition from fossil fuels to renewable energy sources, electrochemistry is necessary in achieving a sustainable future.

Learning Goals

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

Laboratory	• Constructing a table of standard potentials • Utilizing a salt bridge
Conceptual	• Galvanic cells • Cell diagrams • Concentration cells • Nernst equation • Cell potential vs. standard cell potential
Data Analysis	• Using the Nernst equation to determine cell potential • Using standard reduction potentials to calculate standard cell potentials

Procedure

You will **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

1. In preparation for the EDTA experiment, each pair of students must fill one vial with approximately 0.5–0.6 g of pure calcium carbonate.
2. Place the uncapped vial in your 150 mL beaker to keep it from spilling and dry it 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.
3. After 1.5 hours, use the beaker tongs to remove your beaker from the oven. Let the beaker cool until it is warm, but safe to handle.
4. Remove the vial from the beaker using your test tube clamp and place the vial 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.

Special Instructions

- Dispose of all solutions on your spot plate by holding it vertically above the mouth of the funnel in the neck of the metal waste disposal container and rinsing it with a brisk stream from your wash bottle.
- The metal waste containers are in the fumehoods. Wash the spot plate more thoroughly under the deionized water faucet and dry it for later use.
- **Excess solutions containing lead, iron, zinc, and copper need to be disposed of in the metal waste containers.**

Stock Chemicals Used

Chemical	Maximum Amount Used
3.0 M Ammonium Sulfate	< 1 mL
0.1 M Copper Nitrate	~6 mL
0.2 M Ferric Nitrate	< 3 mL
0.1 M Lead Nitrate	~3 mL
0.1 M Sodium Nitrate	~20 mL
0.1 M Zinc Nitrate	1 mL
0.2 M Ferrous Sulfate in 0.1 M Sulfuric Acid	2 mL
Calcium Carbonate *(Solid)*	0.5–0.6 grams

Safety First

Wear your goggles and gloves throughout this experiment.

Treat the voltmeters with care, as they are expensive.

Part I. Constructing a Table of Standard Reduction Potentials

In this part of the experiment, you will be measuring the voltages of the several galvanic cells diagrammed below. Note that the meter display has both an algebraic sign and a magnitude. Throughout this experiment you should clip the "red" wire coming out of the meter to the "right hand" electrode of the cell in question. If the meter shows a "plus" voltage, reduction takes place at that electrode. If the displayed voltage is negative, oxidation is what really occurs at the "right hand" electrode of the cell as diagrammed.

Note that the Fe^{2+} stock solution contains sulfuric acid and some iron nails. This is to ensure that any ferric ions produced by air oxidation are reduced back to the 2+ oxidation state. Because oxygen does attack Fe^{2+}, you should measure the voltages of cells containing the ferrous ion as soon as possible after the solution in question has been prepared. These cell voltages will probably change over time.

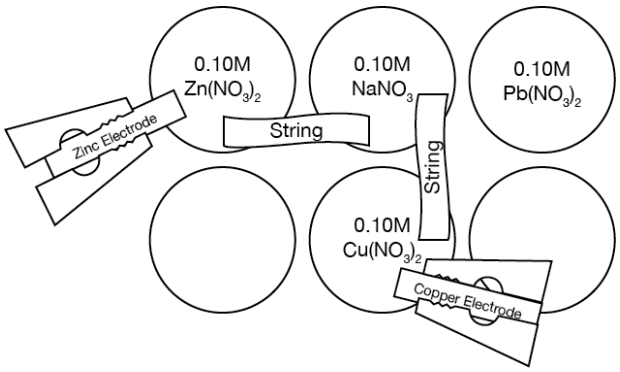
The electrode "compartments" are simply the 1 mL wells in a "spot plate." Fill the wells with a disposable transfer pipet.

The "salt bridge" consists of a well containing 0.1 M sodium nitrate solution connected to two other wells with short (ca. 3 cm) cotton string pre-saturated with 0.1 M $NaNO_3$ solution. You cannot connect the anode and cathode wells directly with the $NaNO_3$ saturated string. You **must** connect both the anode and the cathode to the well containing the 0.1 M $NaNO_3$ solution with the saturated string.

Use the plastic blue tongs provided to manipulate the strings. You should always fill all the wells in the spot plate first and then put the conducting strings in place, because the strings can actually function as siphons.

1. Acquire 10 mL of 0.1 M sodium nitrate solution and soak the stings thoroughly with the solution. Transfer 1 mL of the solution to a well on the spot plate.

2. Assemble and measure the voltages of the following conventional cells. See diagram below for a sample layout for Cell #1:



Cell #1

$$Zn_{(s)} \mid Zn(NO_3)_{2(aq)}\ (0.10\ M) \parallel Cu(NO_3)_{2(aq)}\ (0.10\ M) \mid Cu_{(s)}$$

Cell #2

$$Pb_{(s)} \mid Pb(NO_3)_{2(aq)}\ (0.10\ M) \parallel Cu(NO_3)_{2(aq)}\ (0.10\ M) \mid Cu_{(s)}$$

Cell #3

$$C_{(graphite)} \mid FeSO_{4(aq)}\ (0.10\ M),\ Fe(NO_3)_{3(aq)}\ (0.10\ M) \parallel Cu(NO_3)_{2(aq)}\ (0.10\ M) \mid Cu_{(s)}$$

Green Chem

Use a plastic dispo pipet to transfer excess metal solutions to the waste container.

Waste disposal is costly. Do not unnecessarily add water to the waste container.

Clean-up

- Dispose of all the copper, lead, iron, and zinc-containing solutions on the spot plate in the metal cation waste container found in the fume hood.

Part II. The Concentration Dependence of Half Cell Potentials

Assemble and measure the voltages of the following conventional cells:

Cell #4

$$Cu_{(s)} \mid Cu(NO_3)_{2(aq)}\ (0.010\ M) \parallel Cu(NO_3)_{2(aq)}\ (0.10\ M) \mid Cu_{(s)}$$

Cell #5

$$C_{(graphite)} \mid FeSO_{4(aq)}\ (0.01\ M),\ Fe(NO_3)_{3(aq)}\ (0.10\ M) \parallel Cu(NO_3)_{2(aq)}\ (0.10\ M) \mid Cu_{(s)}$$

Hint

It may be helpful to draw a diagram of your cells.

Some of these cells may be difficult to construct. If you have trouble, consult your TA.

- Prepare 10 mL of 0.02 M $FeSO_4$ by diluting 1 mL of 0.2 M $FeSO_4$ stock solution.
- Mix 1 mL of 0.02 M ferrous sulfate with 1 mL of 0.2 M ferric nitrate for the left hand cells.

Cell #6

$$C_{(graphite)} \mid FeSO_{4(aq)}\ (0.10\ M),\ Fe(NO_3)_{3(aq)}\ (0.010\ M) \parallel Cu(NO_3)_{2(aq)}\ (0.10\ M) \mid Cu_{(s)}$$

Prepare 10 mL of 0.02 M $Fe(NO_3)_3$ by diluting 1 mL of 0.2 M $Fe(NO_3)_3$

- Mix 1mL of the 0.02 M ferric nitrate solution with 1 mL of the 0.2 M ferrous sulfate for the left hand cell.

Clean-up

- Dispose of all the copper, lead, iron, and zinc-containing solutions on the spot plate in the metal cation waste container found in the fume hood.

Part III. Estimating the Solubility Product of Lead(II) Sulfate

For this part of the experiment prepare a 0.050 M $Pb(NO_3)_2$ solution by diluting the stock 0.1 M $Pb(NO_3)_2$ solution by a factor of 2 in one test tube. In another test tube mix equal volumes of 0.10 M $Pb(NO_3)_2$ and 3.0 M ammonium sulfate. Allow about two minutes for the complete precipitation of $PbSO_4$. Assemble and measure the voltage of the cell:

Cell #7

$$Pb_{(s)} \mid PbSO_{4(s)}, NH_4^+ (3.0\ M), SO_4^{2-} (1.5\ M), NO_3^- (0.10\ M), Pb^{2+} (?\ M) \parallel Pb(NO_3)_2\ (0.050\ M) \mid Pb_{(s)}$$

(This is experimentally very easy to do: fill one well of your spot plate with the diluted lead nitrate solution, fill an adjacent one with a slurry of the contents of the test tube containing the precipitated lead sulfate, connect them with salt bridge strings to a well full of the sodium nitrate solution, attach two Pb electrodes to the wires from the meter, and measure the cell voltage.)

Clean-up

- Dispose of all the copper, lead, iron, and zinc-containing solutions in the metal cation waste container found in the fume hood.

Green Chem

Use a plastic dispo pipet to transfer excess metal solutions to the waste container.

Waste disposal is costly. Do not unnecessarily add water to the waste container.

Data Analysis

Part I.

For every conventional galvanic cell diagram there is a corresponding (chemical) cell reaction, as illustrated by the example in the Introduction section of this experiment. According to the Nernst equation the conventional cell voltage of such a cell can be written in general as

$$E_{cell} = (E°_{cathode} - E°_{anode}) - \frac{0.0257}{n} \cdot \ln(Q)$$

where $E°_{cathode}$ is the standard reduction potential of the cathode half-cell reaction and $E°_{anode}$ is the standard reduction potential of the anode half-cell reaction. Q is the reaction quotient, for the overall cell reaction, using the concentrations given in the cell diagram. Note that at equilibrium when Q = K then E_{cell} is equal to zero.

For the cell discussed in the introduction, this equation becomes:

$$\begin{aligned} E_{cell} &= \left(E°_{\frac{Ag+}{Ag}} - E°_{\frac{Cu++}{Cu}}\right) - 0.0129 \ln\left(\frac{0.10M}{0.10M^2}\right) \\ &= (0.799 - 0.337) - 0.0297 \\ &= +0.432V \end{aligned}$$

The numerical values of the standard reduction potentials used in this example were taken from a table in the Appendix of your textbook.

In part I you measured the voltages of three cells, each containing a different metal, against a common Cu(II)/Cu reference electrode. The standard reduction potential of the Cu(II)/Cu*(s)* redox couple is known to be 0.34 V. This value is used as the "known" standard reduction potential in the following calculations.

For each of the three cells whose voltages you measured in Part I of this experiment write the corresponding redox reaction in the spontaneous direction and calculate the value of the standard cell potential, ($E°_{cathode}$ - $E°_{anode}$) using your measured cell potentials and the Nernst equation. Using the standard cell potential you just calculated and the known value of the standard reduction potential of the Cu(II)/Cu*(s)* redox couple, determine the standard reduction potential of each of the noncopper metal half cells. You should also compare your findings with the accepted values.

Part I Analysis.

The following series of questions and calculations will lead you through the calculation of the standard reduction potentials of the half-cells, $Zn^{2+}/Zn_{(s)}$, $Pb^{2+}/Pb_{(s)}$, and Fe^{3+}/Fe^{2+}.

1. Given the Nernst equation, $E_{cell} = (E°_{cathode} - E°_{anode}) - [0.0257/n]lnQ$, and the equation, $E°_{cell} = E°_{cathode} - E°_{anode}$, substitute $E°_{cell}$ into the Nernst equation and solve for $E°_{cell}$. What is the resulting expression for $E°_{cell}$?

2. What is the spontaneous overall cell reaction for the cell $Zn_{(s)} \mid Zn(NO_3)_2$ (0.10 M) $\| Cu(NO_3)_2$ (0.10 M) $\mid Cu_{(s)}$?

3. What is the value of E_{cell} you measured in volts for the spontaneous cell reaction for $Zn_{(s)} \mid Zn(NO_3)_2$ (0.10 M) $\| Cu(NO_3)_2$ (0.10 M) $\mid Cu_{(s)}$?

4. What is the expression for *Q*, the reaction quotient in the Nernst equation, for the spontaneous overall cell reaction in the cell $Zn_{(s)} \mid Zn(NO_3)_2$ (0.10 M) $\| Cu(NO_3)_2$ (0.10 M) $\mid Cu_{(s)}$?

5. Using your cell concentrations and the reaction quotient expression, calculate the value of *Q* for the Nernst equation for the cell $Zn_{(s)} \mid Zn(NO_3)_2$ (0.10 M) $\| Cu(NO_3)_2$ (0.10 M) $\mid Cu_{(s)}$.

6. Calculate the value of $E°_{cell}$ for the cell $Zn_{(s)} \mid Zn(NO_3)_2$ (0.10 M) $\| Cu(NO_3)_2$ (0.10 M) $\mid Cu_{(s)}$ using the value of E_{cell} you measured for the spontaneous reaction and the value you calculated for Q.

7. From the table in your text for the standard electrode potential, E°, what is the accepted value for the half-cell reaction for copper: $Cu^{2+}_{(aq)} + 2e^- \rightarrow Cu_{(s)}$ in volts? You will use this value as a reference to calculate the standard electrode potentials for the other half-reactions involved in the cells you measured.

8. Using the equation $E°_{cell} = E°_{cathode} - E°_{anode}$, and the reference value of E° for $Cu(II)/Cu_{(s)}$ for the role that copper plays in this overall cell reaction, determine the value of the standard potential for the half-reaction $Zn^{2+}_{(aq)} + 2e^- \rightarrow Zn_{(s)}$.

9. What is the spontaneous overall cell reaction for the cell $Pb_{(s)} \mid Pb(NO_3)_2$ (0.10 M) $\| Cu(NO_3)_2$ (0.10 M) $\mid Cu_{(s)}$?

10. What is the value of E_{cell} you measured in volts for the spontaneous cell reaction for cell $Pb_{(s)} \mid Pb(NO_3)_2$ (0.10M) $\| Cu(NO_3)_2$ (0.10M) $\mid Cu_{(s)}$?

11. What is the expression for *Q*, the reaction quotient in the Nernst equation, for the spontaneous overall cell reaction in the cell $Pb_{(s)} \mid Pb(NO_3)_2$ (0.10 M) $\| Cu(NO_3)_2$ (0.10 M) $\mid Cu_{(s)}$?

12. Using your cell concentrations and the reaction quotient expression, calculate the value of *Q* for the Nernst equation for the cell, $Pb_{(s)} \mid Pb(NO_3)_2$ (0.10 M) $\| Cu(NO_3)_2$ (0.10 M) $\mid Cu_{(s)}$.

13. Calculate the value of $E°_{cell}$ for the cell $Pb_{(s)} \mid Pb(NO_3)_2$ (0.10 M) $\| Cu(NO_3)_2$ (0.10 M) $\mid Cu_{(s)}$ using the value of E_{cell} you measured for the spontaneous reaction and the value you calculated for Q.

14. Using the equation $E°_{cell} = E°_{cathode} - E°_{anode}$, and the reference value of E° for Cu(II)/Cu*(s)* for the role that copper plays in this overall cell reaction, determine the value of the standard potential for the half-reaction $Pb^{2+}(aq) + 2e^- \rightarrow Pb(s)$.

15. What is the spontaneous overall cell reaction for the cell C*(gr)* | $Fe(NO_3)_3$ (0.10 M), $FeSO_4$ (0.10 M) || $Cu(NO_3)_2$ (0.10 M) | Cu*(s)*?

16. What is the value of E_{cell} you measured in volts for the spontaneous cell reaction for cell C*(gr)* | $Fe(NO_3)_3$ (0.10 M), $FeSO_4$ (0.10 M) || $Cu(NO_3)_2$ (0.10 M) | Cu*(s)*?

17. What is the expression for *Q*, the reaction quotient in the Nernst equation, for the spontaneous overall cell reaction in the cell C*(gr)* | $Fe(NO_3)_3$ (0.10 M), $FeSO_4$ (0.10 M) || $Cu(NO_3)_2$ (0.10 M) | Cu*(s)*?

18. Using your cell concentrations and the reaction quotient expression, calculate the value of *Q* for the Nernst equation for the cell, C*(gr)* | $Fe(NO_3)_3$ (0.10 M), $FeSO_4$ (0.10 M) || $Cu(NO_3)_2$ (0.10 M) | Cu*(s)*.

19. Calculate the value of $E°_{cell}$ for the cell C*(gr)* | $Fe(NO_3)_3$ (0.10 M), $FeSO_4$ (0.10 M) || $Cu(NO_3)_2$ (0.10 M) | Cu*(s)* using the value of E_{cell} you measured for the spontaneous reaction and the value you calculated for Q.

20. Using the equation $E°_{cell} = E°_{cathode} - E°_{anode}$, and the reference value of E° for Cu(II)/Cu*(s)* for the role that copper plays in this overall cell reaction, determine the value of the standard potential for the half-reaction $Fe^{3+}(aq) + e^- \rightarrow Fe^{2+}(aq)$.

Part II.

Write the chemical reaction in the spontaneous direction for the copper concentration cells—the first cell you examined in Part II of the experiment. Calculate the theoretical value of the potentials for these copper concentration cells using the Nernst equation. Compare the measured and theoretical values for these cell potentials. Your agreement will be only semi-quantitative.

Altogether, you have measured the voltages of three cells with different relative concentrations of Fe^{2+} and Fe^{3+} ions against a common Cu^{2+}/Cu reference electrode. If you write out the Nernst equation for these three cells and expand the logarithmic terms, you will find that in each case the cell voltage can be written as a sum of four terms. [Hint: $\ln (a \cdot b / c) = (\ln a) + (\ln b) - (\ln c)$] Three of them are invariant and include the E°'s of the iron and copper half reactions along with the term that takes the log of the copper ion concentration into account. The fourth term, however, varies in value and involves only the log of the ratio of the iron species concentrations, and must therefore be responsible for the voltage changes you observed in the laboratory. Calculate the theoretical cell voltage DIFFERENCES for the three concentration cells involving the copper and iron and compare your experimental results with these predictions. Again you should expect only semi-quantitative agreement. In separate calculations, find the cell voltage difference between cell #3 in Part I and each of the iron containing cells of Part II.

Part II Analysis.

1. Using the facts that a spontaneous cell reaction gives a positive measured voltage when the red lead of the voltmeter is connected to the cathode, and that reduction takes place at the cathode while oxidation takes place at the anode, what is the spontaneous reaction for the copper concentration cell you measured in Part II of the electrochemistry experiment?

2. Because the standard potentials are the same for both the anode and the cathode reactions for a concentration cell, the Nernst equation for a concentration cell becomes simply $E_{cell} = -(0.0257/n)\ln Q$, where *Q* is to be calculated for the spontaneous cell reaction. What is the value of *Q* for the copper concentration cell?

3. $E_{cell} = -(0.0257/n)\ln Q$. Now, using your value for Q, calculate E_{cell} from the Nernst equation for the copper concentration cell.

4. For Part II of the electrochemistry experiment, you measured the potential of some concentration cells. The laboratory manual asked you to construct the cells

   a) $Cu_{(s)}|Cu(NO_3)_2$ (0.010 M) || $Cu(NO_3)_2$ (0.10 M) | $Cu_{(s)}$

   b) $C_{(graphite)}$ | $FeSO_4$(0.010 M), $Fe(NO_3)_3$ (0.10 M) || $Cu(NO_3)_2$(0.10 M) | $Cu_{(s)}$

   c) $C_{(graphite)}$ | $FeSO_4$ (0.010 M), $Fe(NO_3)_3$(0.10 M) || $Cu(NO_3)_2$(0.10 M) | $Cu_{(s)}$

   When the red lead for the voltmeter was connected to the right-hand electrode of the cells as diagrammed above, which of these cells showed a positive algebraic sign for the measured voltage?

5. When a measured cell voltage is negative, the electrode to which the red lead is attached is actually the anode rather than the cathode. The anode is the electrode at which oxidation takes place, so it is surrendering electrons to the external circuit. For the two cells involving the iron and copper species, the Cu(II)/$Cu_{(s)}$ couple is the anode. What half-cell reactions is taking place at the Cu electrode in the anode compartment of these two cells?

6. Calculate the theoretical cell voltage differences for the three concentration cells involving the copper and iron, cells #3, #5, and #6.

7. What is the expression for *Q*, the reaction quotient in the Nernst equation for the overall cell reaction in cells #3, #5, & #6, $Cu_{(s)} + 2Fe^{3+}_{(aq)} \rightarrow 2Fe^{2+}_{(aq)} + Cu^{2+}_{(aq)}$?

8. The Nernst equation for the cell potential of cells #3, #5, & #6, is

   $E_{cell} = E°_{cell} - (0.0257/2)\ln(Q_{\text{previous question}})$.

   What is the expression, then, for the cell voltage difference between cells #5 and #3, E_{cell}#5 - E_{cell}#3?

9. Using the ion concentration values in cells #3 & #5 in the expression obtained from the previous question, calculate the theoretical cell voltage difference between cells #5 and #3.

10. Using the ion concentration values in cells #3 & #6 and the expression for E_{cell}#6 - E_{cell}#3, calculate the theoretical cell voltage difference between cells #6 and #3.

Part III.

In this experiment, what you have really done is prepare a concentration cell with drastically different concentrations of the Pb^{2+} ion in the two halves of the cell. Use the Nernst equation and the measured cell voltage to estimate the Pb^{2+} ion concentration in the compartment containing the lead sulfate slurry. The solubility product of lead sulfate can then be estimated by multiplying this result by the concentration of the sulfate ion in that compartment. Your result should be within about a factor of ten of the tabulated value for this constant.

Part III Analysis.

1. Calculate the solubility product for $PbSO_4$ by using your measured potential for cell #7 in Part III of the laboratory.

2. What is the value of E_{cell} you measured in volts for the spontaneous cell reaction for cell $Pb_{(s)}$ | $PbSO_{4(s)}$, NH_4^+ (3.0 M), SO_4^{2-} (1.50 M), NO_3^- (0.10 M), Pb^{2+}(? M) || $Pb(NO_3)_2$ (0.050 M) | $Pb_{(s)}$?

3. Because the standard potentials are the same for both the anode and the cathode reactions for a concentration cell, the Nernst equation for a concentration cell becomes simply $E_{cell} = -(0.0257/n)\ln Q$, where *Q* is the expression for the spontaneous cell reaction. For the concentration cell, $Pb_{(s)}$ | $PbSO_{4(s)}$, NH_4^+ (3.0 M), SO_4^{2-} (1.50 M), NO_3^- (0.10 M), Pb^{2+} (? M) || $Pb(NO_3)_2$ (0.050 M) | $Pb_{(s)}$, what is the expression for *Q* for the spontaneous overall cell reaction, Pb^{2+} (0.050M) → Pb^{2+} (?)?

4. For the concentration cell, Pb(s) | $PbSO_4$(s), NH_4^+ (3.0 M), SO_4^{2-}(1.50 M), NO_3^- (0.10 M), Pb^{2+}(? M) || $Pb(NO_3)_2$(0.050 M) | Pb(s), $E_{cell} = -(0.0257/2)\ln(Q_{previous\ question})$, what is the expression for *x* in terms of E_{cell}?

5. Using your measured cell potential for $Pb_{(s)}$ | $PbSO_{4(s)}$, NH_4^+ (3.0 M), SO_4^{2-} (1.50 M), NO_3^- (0.10 M), Pb^{2+} (? M) || $Pb(NO_3)_2$ (0.050 M) | $Pb_{(s)}$ in your expression for *x*, calculate the molarity of Pb^{2+} in the anode compartment.

6. What is the solubility product expression, K_{sp}, for $PbSO_4$?

7. Using the expression, $K_{sp} = [Pb^{2+}][SO_4^{2-}]$, the concentration of sulfate in the anode compartment, and the determined value of Pb^{2+} concentration in the anode compartment, calculate K_{sp}.

EDTA Titrations

Introduction

In these times of environmental awareness and concern, it is very important that you become experienced with water analysis. As you know, Davis is not known for the exceptional quality of its water. In fact, let's face it, the water here tastes pretty bad! What is in it? That would be a good question to try to answer if we had lots of time and if we wanted to spend it doing an experiment in qualitative analysis. However, for now we want to learn more about *quantitative analysis*. It is well known that ground water commonly contains a large amount of two metal ions, calcium and magnesium. The term "hard water" refers to the presence of these two metals. These metals give the water a rather harsh taste and will also cause the white deposits often observed on faucets or as "lime" deposits in bathtubs. These white deposits are generally metal carbonates. In addition, these two metal ions will precipitate soaps, leaving the unsightly bathtub scum that you may have observed. Perhaps you have had the experience of living or visiting a residence where the water has been "softened". Softening refers to a process whereby the water is passed through a column in which the calcium and magnesium ions are removed. This makes the water feel "softer" when taking a bath or shower, because the soap precipitates do not form.

In this experiment, you will determine the amount of calcium carbonate present in an unknown solid sample the stockroom has prepared. To do this you will use a hexadentate ligand called ethylenediaminetetraacetic acid (EDTA). This ligand, due to its six donor atoms and to its size and shape, has the exceptional ability to complex or chelate with a variety of metal ions. The equilibrium constant for the formation of each metal/EDTA complex is different, and the kinetic rate at which each metal complexing agent forms is different as well. Thus, the ligand can be used to complex one metal ion in the presence of another. For example, calcium-EDTA is given as an antidote for mercury ion poisoning. Once the EDTA is ingested, it will selectively bind to the free mercury ions, effectively removing them and not allowing them to bind to enzymes and cytochromes. Thus the poison is removed and passes innocuously through the physiological system. For our analysis, we will use disodium EDTA to bind to calcium and magnesium ion as is shown below:

$$2OH^- + Ca^{2+} + H_2EDTA^{2-} \rightarrow CaEDTA^{2-} + 2H_2O \qquad K = 5.0 \times 10^{10}$$

$$2OH^- + Mg^{2+} + H_2EDTA^{2-} \rightarrow MgEDTA^{2-} + 2H_2O \qquad K = 5.0 \times 10^{8}$$

However, we need something to indicate when the reaction is complete. We need an indicator. The indicator will need to be another ligand and it must have a different color when it is free than when bound to a metal ion. One such ligand is Calmagite that binds to alkaline earth metals producing a color change as follows:

$$M^{2+} + In^{2-}\text{ (blue)} \rightarrow MIn\text{ (red or pink)} \qquad K = \sim 7 \times 10^{-9}$$

The reaction of EDTA with metals ($K \sim 10^8$) is greater than that of Calmagite ($K \sim 10^{-9}$). Thus, if a small amount of indicator is added to a solution of magnesium and calcium, a red or pink colored complex will result. If EDTA is then added via a buret, the color will change when the metal

is stripped from the Calmagite and binds to the EDTA; the solution will turn blue. If we have carefully measured the amount of EDTA that we have added, then we can determine the total amount of calcium and/or magnesium in the sample.

It is important that you appreciate that EDTA has acid/base properties. It has four acid constants, as is shown below (Note: $Y = EDTA^{4-}$)

$$H_4Y \rightarrow H^+ + H_3Y^- \qquad K_{a1} = 1.0 \times 10^{-2}$$

$$H_3Y^- \rightarrow H^+ + H_2Y^{2-} \qquad K_{a2} = 2.2 \times 10^{-3}$$

$$H_2Y^{2-} \rightarrow H^+ + HY^{3-} \qquad K_{a3} = 6.9 \times 10^{-7}$$

$$HY^{3-} \rightarrow H^+ + Y^{4-} \qquad K_{a4} = 5.5 \times 10^{-11}$$

For this reason, analyses must be done at a constant pH and one that will enable the ligand to bind successfully with the metal. These determinations will be conducted at pH 10 via the addition of a NH_4OH/NH_4Cl ($pK_a = 9.24$) buffer.

Toxic Lead (Pb) Poisoning and EDTA Chelation Therapy

In the spring of 2014, residents of Flint, Michigan started raising concerns about their water. They noticed a strange smell, an odd taste, discoloration (turning orange), and the subsequent illnesses among children. It was later discovered that the water was contaminated with chloride, iron, and a toxic level of lead. To address such poisonings, one accepted treatment is the use of Ethylenediaminetetraacetic Acid (EDTA) chelation therapy. EDTA works by capturing heavy metal ions in the bloodstream, forming a compound that can be eliminated from the body in as little as 1-3 hours.

The incident described above became widely known as the Flint Water Crisis and remains the largest level of lead contamination to be found in the U.S. drinking water to date. The question arises: how did it happen? In early 2013, officials in Flint made the decision to switch the city's water supply source from the Detroit River to the Flint River and transitioned from a Detroit-based water treatment plant to a Flint-based water treatment plant. Unfortunately, this change immediately led to water-related problems due to various chemical reactions occurring in the pipelines used for water distribution. It's important to note that at that time, water pipelines were often made of lead (Pb), and the majority of Flint's water pipelines were composed of this material. The issue only arose because of the new water source and its treatment.

There were three main reasons for the significant increase in Pb levels:

1. Unlike the Detroit plant, the Flint water treatment plant did not treat its water with a phosphate corrosion inhibitor. This allowed for oxidants and chloride ions to accelerate corrosion in the pipes.

2. The Flint River, the new water source, had an increased amount of chloride ions, which led to further increased corrosion in the pipes.

3. The final and perhaps the most critical factor was that the water treated in Flint had a lower pH than the water treated in Detroit. This lower pH caused the dissolution of the carbonate inhibitor, which acts as the last safeguard by lining the inside of lead pipes and prevents them from corroding.

It is important to note that nearly everyone reading this information has consumed water transported through lead pipes at some point. However, it took a combination of these three issues to lead to the severity of the Flint Water Crisis.

Learning Goals

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

Laboratory	• Preparing a standard • Performing a titration • Using an indicator
Conceptual	• Reaction equilibrium (K_{eq}) • Polyprotic acid constants (K_a) for EDTA • Binding of polydentate ligands to metal ions, specifically hexadentate ligand EDTA
Data Analysis	• Determining unknown concentration of unwanted ions

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.

Safety First

The pH 10 buffer is 10 M ammonia. Keep it under the fume hood as much as possible and avoid breathing its vapors.

Wear your goggles!

Stock Chemicals Used

Chemical	Maximum Amount Used
6 M Hydrochloric Acid	< 6 mL
EDTA Disodium Salt (dihydrate), *(Solid)*	< 10 grams
Calcium Carbonate, *(Solid)*	0.5–0.6 grams
Calmagite Indicator	< 3 mL
Magnesium EDTA solution	6 mL
pH 10 Ammonia Buffer	50 mL

Pre-Laboratory Preparation

During the laboratory period before beginning this experiment (Electrochemical Cells), you were instructed to dry a sample of pure calcium carbonate.

Part I. Preparation and Standardization of an EDTA Solution

Hint

Calculate the mass of disodium ethylene-diaminetetraacetate dihydrate **(CAS 6381-92-6)** needed for the preparation of your stock EDTA solution.

1. Prepare approximately 250 mL of a 0.06 M Na_2EDTA solution by filling a 250 mL Erlenmeyer flask with approximately 150 mL of deionized water. Add the necessary mass of solid $Na_2EDTA{\cdot}2H_2O$. Then, take your solution to the fume hood and add about 20 mL of the pH 10 buffer. Swirl the flask carefully to dissolve. Mix well and add deionized water to your EDTA solution to make approximately 250 mL of solution.

2. Prepare a calcium carbonate standard solution for the standardization of your EDTA solution.

    - Measure approximately 0.45 g $CaCO_3$. Record the mass to the nearest mg.
    - Transfer the solid $CaCO_3$ to a 100 mL beaker
    - Dropwise add 6 M HCl to dissolve the $CaCO_3$. Do not over acidify. It should take no more than 2 mL.
    - Observe the evolution of CO_2 gas as the carbonate reacts with HCl. Wait for the $CaCO_3$ to dissolve completely before proceeding to the next step.

- Quantitatively transfer the dissolved calcium carbonate solution to a 25 mL volumetric flask. Using a water bottle, add DI water to the volumetric flask until the volume reaches 25.00 mL.

3. Transfer one 5.00 mL aliquot of the $CaCO_3$ solution to a 125 mL or 250 mL Erlenmeyer flask.

Question A: Write the balanced chemical equation for the standardization of EDTA solution.

4. It is absolutely essential that the pH of the calcium carbonate solution remains at 10 throughout the titration. To ensure that the pH remains at 10, take the Erlenmeyer flask with calcium carbonate and beaker with 30 mL of water to the fume hood.

5. Add 5 mL of the pH 10 ammonia buffer and then the 30 mL of water to the Erlenmeyer in the fume hood. Only after you have added these 2 chemicals to your Erlenmeyer flask, can you leave the fume hood and return to your bench to check the pH of the solution using Alkacid paper. You may also want to check the pH of the solution a couple of times during the titration.

6. Five to six drops of Calmagite indicator is sufficient to show the color change at the endpoint. One can also sharpen the endpoint by adding about 1 mL of the solution labeled $Na_2MgEDTA$.

   The magnesium ion is approximately 40 times more strongly bound to the indicator Calmagite than the calcium ion. Moreover, the calcium ion is approximately 200 times more tightly bound to the ligand EDTA. Thus, when EDTA is added to the solution, it will preferentially bond to the calcium ion.

   When the calcium ion has completely reacted, the EDTA will then pull the magnesium ion away from the indicator resulting in a color change of the solution. Note that adding one mL of this solution does not affect the stoichiometry of the titration as the solution contains an equal molar amount of magnesium and EDTA.

   Be sure to not stop titrating when the solution is the purple color, and keep titrating all the way to the **blue color endpoint**.

7. Keep the flask of your first trial titration to use as a reference color for subsequent trials. Be sure you have three acceptable trials before moving on to Part II.

   To determine if a trial is acceptable, calculate the molarity of the EDTA solution based on your volumes and mass of $CaCO_3$ for each trial and then perform the Q-test. For more details regarding the Q-test calculation, see the Appendix section of your laboratory manual.

Hint

Do not use the buffer solution found on the shelves in the lab. **Add the 10 M ammonia buffer found in the fume hood.**

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.

Question B: Write the balanced chemical equation for the reaction between the EDTA solution and the indicator, MgIn*(aq)*.

8. Perform the analysis with three samples. Calculate the molarity of the EDTA solution for each sample. Calculate an average molarity and the standard deviation. The post-lab exercises will guide you through these calculations.

Part II. Determination of Calcium in an Unknown

1. Clean three 125 mL, 250 mL or 500 mL Erlenmeyer flasks. It is very important that the flasks be extremely clean and well rinsed with deionized water. Accurately weigh three samples of your dry unknown into the three Erlenmeyer flasks. The unknown samples should weigh between 0.150–0.180 grams.

Note

Remember to record your unknown number before you leave the lab!

You will need to know your unknown number for the post-lab analysis.

2. Prepare and titrate the unknown samples using a procedure similar to the one you used for the standardization of your EDTA solution.

    - Dissolve your unknown by adding 6 M HCl dropwise. Do not over acidify. It should take no more than 2 mL.
    - Observe the evolution of CO_2 gas as the carbonate reacts with HCl. Wait for the unknown to dissolve completely before proceeding.
    - In a fume hood, add 5 mL of pH 10 ammonia buffer and 30 mL of deionized water to your unknown sample. Return to your bench to check the pH of your sample.
    - Add five to six drops of Calmagite indicator. Add no more than 1 mL of $Na_2MgEDTA$.
    - Titrate your unknown using your EDTA solution.

    Be sure you have three acceptable trials before cleaning up. To determine if a trial is acceptable, calculate the percent mass $CaCO_3$ in the sample for each trial based on your volumes of EDTA and mass of $CaCO_3$ for each trial and then perform the Q-test.

    Report the average percent by mass of $CaCO_3$ in your unknown along with both a relative and standard deviation, and a 90% confidence limit. The post-lab exercises will guide you through these calculations.

Clean-up

- Rinse all glassware with deionized water.
- Return all chemicals and equipment to the proper location. All solutions may go down the drain with copious amounts of water.
- Rinse the bench with your sponge and water.

Data Analysis

Part I. Standardization of EDTA solution

1. In the standardization of the sodium EDTA solution using the solid calcium carbonate primary standard, what is the precise mass in **GRAMS** of the dry calcium carbonate you used to prepare the primary standard solution? Your mass precision should be reported to a thousandth of a gram.
2. Using the mass of calcium carbonate you used and the volume of dilution, determine the molarity of Ca^{2+} in the primary standard solution.
3. For each of your trials, what is the precise volume in mL of the standard calcium solution used in the standardization of the sodium EDTA solution?
4. For each of your trials, what is the precise volume in mL of the sodium EDTA solution used in the titration of your standard calcium solution?
5. For each of your trials, calculate the molarity of your EDTA solution using the molarity of your standard calcium solution, the volume of standard calcium solution used, and the volume of EDTA used in the titration of your standard calcium solution.
6. Calculate the average molarity of your EDTA solution.
7. Calculate the standard deviation of the molarities of the EDTA solution.

Part II. Determination of Calcium in an Unknown

8. For each of your trials, what is the mass of your unknown sample in grams?
9. In the titration of the dry unknown sample with the secondary standard solution, EDTA, your titration volumes of the EDTA solution should be approximately 10-15 mL. For each of your trials, what is the precise volume in **milliliters** of the EDTA solution used in the titration of your unknown sample (e.g. 11.81 mL).
10. Using the volume of EDTA solution, the mass of unknown sample, and the average molarity of the EDTA solution, calculate the percent mass of calcium carbonate in the dry unknown sample for each of your trials.
11. Calculate the average mass percent of calcium carbonate in your dry unknown sample.
12. Calculate the standard deviation of the mass percent of calcium carbonate in your dry unknown sample.
13. Calculate the 90% confidence limit for the average percent mass of the calcium carbonate in your dry unknown sample.
14. Your TA has assigned you an Unknown Number between 1-12 for your Calcium Carbonate unknown. Record your Unknown Number.

Introduction to Inorganic Qualitative Analysis

Introduction

In this experiment you will be introduced in an abbreviated way to systematic methods that chemists have traditionally used to identify the cationic constituents of a mixture. In most such schemes, including this one, a sequence of precipitating agents is used to separate the original mixture into smaller groups, each of which may contain more than one constituent. Each of the smaller groups is then examined further.

Modern Techniques for Metal Cation Detection

Inorganic qualitative analysis serves as a fundamental building block in scientific understanding of metal cation detection in aqueous solutions. Without the knowledge provided by this method, the development of more advanced techniques would not have been possible. Atomic Absorption Spectroscopy (AAS) and Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) are modern methods used for metal cation detection. These techniques have now replaced qualitative analysis due to their superior speed, precision, and sensitivity. Moreover, they possess the remarkable capability of identifying multiple metal cations within a single sample. However, one of the major drawbacks to these methods is their cost. They are expensive, with prices that can range from $50,000 to $500,000.

The elements that you will be focusing on are seven of the twelve metallic constituents of the third long row of the periodic table: K, Ca, Cr, Mn, Fe, Co, and Zn. You will first encounter them as 0.05 M aqueous solutions of nitrate salts. During the course of the experiment some of these elements may change their oxidation states one or more times.

The scheme you will be using does not include the Cu^{2+} ion because it behaves rather unpredictably when subjected to the reagents used here to effect the separation of a mixture of these cations. In more elaborate analytical schemes the copper gets removed early on by precipitating it as the sulfide from a 0.3 M acid solution saturated with hydrogen sulfide. The sulfide ion concentration in such a solution is so incredibly low that only CuS ($K_{sp} \sim 10^{-35}$) precipitates at that point.

The first part of the experiment comprises a series of diagnostic tests in which you will determine experimentally how five of the seven salts respond to the various treatments that will comprise the overall analytical scheme. *These tests may all be performed in a single laboratory period if you have thoroughly studied the experiment before you come to lab so that you know exactly what you will be doing and in what order.*

Learning Goals

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

Laboratory	• Separations of solutions • Neutralizing reactions • Importance of clean glassware • Using a centrifuge
Conceptual	• Solubility rules • Replacement reactions • Redox reactions • Complex ion reactions • Equilibrium
Data Analysis	• Replacement reactions

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 Nitric Acid	< 5 mL
6 M Sodium Hydroxide	< 5 mL
30% Hydrogen Peroxide	1 mL
0.05 M Calcium Nitrate	1 mL
0.05 M Chromium Nitrate	1 mL
0.05 M Cobalt Nitrate	1 mL
0.05 M Ferric Nitrate	1 mL
0.05 M Manganese Nitrate	1 mL
0.05 M Potassium Nitrate	1 mL
0.05 M Zinc Nitrate	1 mL
0.1 M Lead Nitrate	A few drops
0.2 M Potassium Oxalate	A few drops
0.1 M Potassium Thiocyanate	2 drops
0.05 M Sodium Tetraphenylborate	2 drops
Thymolphthalein Indicator	A few drops
Sodium Bismuthate, (Solid)	~0.2 grams
Potassium Nitrite, (Solid)	0.2 grams
4 M Potassium Acetate	< 2 mL
0.05% Dithizone in Chloroform	3 drops
6 M Acetic Acid	< 2 mL
Acetone Wash Bottle	< 25 mL

Safety First

Wear your goggles and gloves throughout this experiment.

Some of the reagents used in this experiment can cause burns or other skin damage.

Part I. Qualitative Analysis—Analysis of a Known Solution

Setup

1. All glassware must be kept scrupulously clean. Each reagent/test solution should have its own dropper.
2. Start a hot water bath in a 400 mL beaker (⅓-full of water).
3. Set aside another beaker to serve as a waste container.

4. Make a 3% H_2O_2 solution. (Dilute the 30% H_2O_2 stock solution by a factor of 10 using deionized H_2O.) Store this solution in a clean test tube and supply with a plastic pipet.

Making the Known Solution

1. To make your known solution, 1 person from each bay (i.e. the collection of 4 bench tops) will combine 1 mL of each of the following metal nitrate solutions in either a 50 mL Erlenmeyer flask or a 100 mL beaker: Ca^{2+}, Co^{2+}, Cr^{3+}, Fe^{3+}, K^{+}, Mn^{2+}, Zn^{2+}. The solution can be mixed by drawing and expelling it with a clean disposable pipet or swirling it in the container it was made in. Once that person returns to the bay with the mixed solution, each group will take 1 mL of the solution and place it in a clean centrifuge tube.
2. Be sure and note the colors of each individual solution in your Lab Notebook.

Initial Separation of the Cr^{3+}, Fe^{3+}, K^{+}, Mn^{2+}, Zn^{2+} Ions

Step 1

To the 1 mL cation solution in the centrifuge tube, you will add the following chemicals to separate the ions:

1. Add 10 drops of 6 M NaOH (shake after every 2 drops.)
2. Add 5 drops of 3% H_2O_2. Shake tube and place in hot water bath for 3 minutes.
3. If a precipitate is present, do the following:
   a. Centrifuge the sample.
   b. Check for complete precipitation (ppt) by adding a few more drops of 6 M NaOH and watching for the additional formation of ppt on the top of the solution.
   c. When ppt is complete, decant the supernatant liquid and SAVE this for Step 5.

Step 2

To the ppt from the previous step,

1. Add 10 drops of 6 M HNO_3.
2. Stir the solution well and place the tube in the hot water bath for 3 minutes.
3. Centrifuge (if necessary) and remove supernatant liquid. SAVE this for Step 4.

Lab Skill Tips

Balance the test tubes before placing them in the centrifuge.

Make sure each pair of opposing test tubes contain similar volumes before starting the centrifuge.

Do not open the centrifuge lid before the rotor **completely** stops.

Confirmation Tests for the Mn^{2+} and Co^{2+} Ions

Step 3

To the ppt from Step 2,

1. Add about 5 drops of 3% H_2O_2 (or until the ppt dissolves).
2. Place in a hot water bath (using a hot plate) for about 3 minutes (or until bubbling stops).
3. Place a small amount of this solution in a spot plate well.
    a. Add a small sprinkle of $NaBiO_3$.
    b. Stir. If a reaction is observed, then the Mn^{2+} cation has been confirmed.
4. To the remaining solution in the tube,
    a. In the fume hood, add 0.2 g of KNO_2(s). Then add a few drops to 1 mL of 4M $KC_2H_3O_2$(aq). The combined volume of KNO_2(s) and $KC_2H_3O_2$(aq) should be approximately equal to the volume of the solution in the tube.
    b. Seal the tube with parafilm and shake vigorously. If a reaction is observed, then the Co^{2+} cation has been confirmed. If NO reaction was observed, don't panic! You will be prompted to try another test for Co^{2+} later.

Confirmation Tests for the Fe^{3+} Ion (and Alternative Test for Co^{2+})

Step 4

To the tube containing the liquid from Step 2,

1. Add 6 M NaOH until a ppt is visible.
2. Then add 1 drop of 6 M HNO_3 until the ppt disappears again. It may be necessary to add more than 1 drop. Your solution is now neutralized.
3. Place a small amount of this solution in a well of a spot plate. Add 2 drops of KSCN. If a reaction is observed, then the Fe^{3+} cation has been confirmed.
4. If the confirmation test for Co^{2+} in Step 3, 4b for did not show a reaction, then
    a. In the fume hood, to the remaining neutralized solution in your tube, add equal volumes of 0.2 g KNO_2*(s)* and 4 M $KC_2H_3O_2$*(aq)*. Again, this should require a few drops to 1 mL of the 4 M $KC_2H_3O_2$*(aq)*.

Safety First

Never walk around with a filled pipet in your hand. Keep dispo pipets in a test tube to reduce accidental spills.

b. Seal the tube with parafilm and shake vigorously. If a reaction is observed, then the Co^{2+} cation has been confirmed.

Confirmation Tests for Cr^{3+} and Zn^{2+} ion

Step 5

To the liquid from Step 1,

1. Add 1 drop of the thymolphthalein indicator.
2. Add 6 M acetic acid dropwise (while shaking gently) until you see some sort of color change.
3. Place a small amount of this solution in a spot plate well.
   a. Add a few drops of 0.1 M $Pb(NO_3)_2$ to the well. If a reaction is observed, then the Cr^{3+} cation has been confirmed.
   b. In the fume hood, add 3 drops of dithizone in chloroform to the remaining solution in the tube. If a reaction is observed, then the Zn^{2+} cation has been confirmed.

   Do not remove this test tube from the fume hood! Pour the contents of the test tube straight into the waste container.

Safety First

Chloroform can cause severe health effects if inhaled. Do not move any containers containing chloroform outside of the fume hood.

Do not take the tube containing dithizone out of the fume hood!

Clean-up

- **In the fumehood**, rinse the tube containing dithizone (using acetone) into the Dithizone Waste Container.
- Any remaining waste can be transferred to your waste beaker.

Confirmation Tests for Ca^{2+} and K^+ ions

Step 6

Place a small amount of the **original** solution in a spot plate well, and in a disposable culture tube.

1. To the solution in the well, add 2 drops of sodium tetraphenylborate. If a reaction is observed, then the K^+ cation has been confirmed.
2. To the solution in the tube, add 5 drops or a small amount of 0.2 M $K_2C_2O_4$. If a reaction is observed, then the Ca^{2+} cation has been confirmed.

Clean-up

- Make sure all waste gets transferred to the appropriate waste containers in the fumehood: Cation Waste or Dithizone/ chloroform/acetone Waste Container.
- Neutralize excess sodium hydroxide with equal volume of nitric acid. Add **0.5 g of sodium bicarbonate** to the mixture and dispose of the neutralized solution with copious amount of water.

Data Analysis

Part I. Qualitative Analysis – Due before beginning Part II. To be completed on-line.

After completing Part I, **please complete the on-line post-laboratory exercises for week 1.** The questions you will answer on-line are shown below as questions 1–7. If you have completed the on-line exercises for Part 1 of this laboratory, your TA will provide you with a blank "flow chart."

Record the correct answers here while you do the on-line exercise and bring these answers to lab class with you.

Question 1. An unknown solution could contain any or all of the following cation: K^+, Ca^{2+}, Cr^{3+}, Mn^{2+}, Fe^{3+}, Co^{2+}, Zn^{2+}. A 1 mL portion of the unknown is treated with 2 mL of 6 M NaOH, added a few drops at a time with shaking. Immediately thereafter 10 drops of 3% hydrogen peroxide are added dropwise with shaking, and the centrifuge tube is then heated for a few minutes in a water bath until bubbling ceases.

The resulting conglomerate is then centrifuged. Check for complete precipitation by adding 6 M NaOH. The supernatant liquid is decanted off and saved. (Although you have not demonstrated these facts for yourself, Ca^{2+} will be precipitated as the hydroxide at this stage and K^+ will remain in the decanted liquid.)

At this stage of the experiment the elements that could be found in the decanted liquid are:

_____ _____ _____ _____ _____ _____ _____

The elements that could be found in the remaining precipitate are:

_____ _____ _____ _____ _____ _____ _____

Question 2. The precipitate is treated with 10 drops of 6 M HNO_3. The centrifuge tube is then heated briefly in a water bath. The centrifuge tube is then centrifuged, and the supernatant liquid is decanted and saved. (The $Ca(OH)_2$ produced in Question 1 is acid soluble.)

At this stage of the experiment the elements that could be found in the decanted liquid are:

_____ _____ _____ _____ _____ _____ _____

The elements that could be found in the remaining precipitate are:

_____ _____ _____ _____ _____ _____ _____

Question 3. The precipitate from Question 2 is treated with 5 drops of 3% hydrogen peroxide until the precipitate fully dissolves. The centrifuge tube is then heated in a water bath until bubbling ceases.

At this stage of the experiment the elements that could be found in the resulting liquid are:

_____ _____ _____ _____ _____ _____ _____

The elements that could be found in the remaining precipitate are:

_____ _____ _____ _____ _____ _____ _____

Question 4. The decantate from Question 1 is neutralized with acetic acid and divided into the appropriate number of portions. How many portions? What should you do with each of them? What would be the observation if each treatment led to a positive (confirmatory) result?

Question 5. The final decantate from Question 2 is divided into the appropriate number of wells of a spot plate and tested as in Part I of this experiment. How many wells? What reagents would be required for each spot-test, and what would be the observation if each spot-test led to a positive (confirmatory) result?

Question 6. The liquid from Question 3 is divided into the appropriate number of portions and again spot tested. What reagents would be required and what would be the observation if each spot-test led to a positive (confirmatory) result?

Question 7. The original unknown solution is tested separately for the presence of potassium and calcium. How will you perform these tests?

Part II. Qualitative Analysis — Analysis of an Unknown Mixture

1. Obtain an unknown cation solution from your TA. The unknown solution contains **5 cations**. You will only receive ~2 mL of unknown. Plan your procedures accordingly.
2. Note the initial color of your unknown solution. How does this compare to the colors of the original known solutions? What cations are likely to be present in your unknown?
3. Record your Unknown # in your Lab Notebook.
4. Follow the same steps as Part I until you have confirmed all **5 unknown cations.**
5. Perform the Final Confirmation for your unknown (see below).

Final Confirmation Steps

1. Once you have determined the 5 cations present in your solution, mix up a 'theoretical unknown solution' containing 1mL of each of the known cations that you think are present in your unknown solution.
   a. Place 1 mL of this solution in a centrifuge tube.
   b. Place 1 mL of your **original** unknown solution in a second centrifuge tube.
2. Compare the color of the 2 solutions from Step 1–2 of Part II.
   a. Are they the same?
3. Run several of the tests described in Part I side-by-side and compare.
   a. Do they behave the same way?
   b. Are the ppt colors exactly the same?
4. If so, then you have confirmed the cations in your unknown solution.

Safety First

Don't unplug or put back the hot plates while the top is still hot—you can tell the top is cool when the Caution light turns off.

Clean-up

- Make sure all waste is transferred to the appropriate waste containers in the fumehood: Cation Waste or Dithizone/ chloroform/acetone Waste Container.
- Neutralize excess sodium hydroxide with equal volume of nitric acid. Add **0.5 g of sodium bicarbonate** to the mixture and dispose of the neutralized solution with copious amount of water.

Complete your post-laboratory exercises for Part II! You will be asked for your unknown # and all the confirmed cations in that unknown.

Synthesis of a Transition Metal Coordination Complex

trans-$[Co(en)_2Cl_2]Cl$

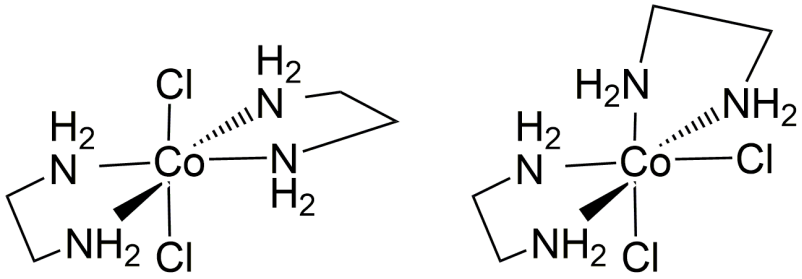
Introduction

In this experiment you will prepare a coordination compound of Cobalt(III) with the bidentate ligand ethylenediamine ("en"; $NH_2CH_2CH_2NH_2$). Coordination compounds are also called complexes, and if they are ions they are called complex ions. A complex ion contains a central metal to which Lewis bases (ligands) have been attached or coordinated. The purpose of this experiment is to provide an introduction to the field of transition metal coordination chemistry and have you synthesize a coordination compound. This synthesis will allow you to experience some of the common procedures used to prepare and isolate inorganic compounds. The complex you will synthesize is *trans*-$[Co(en)_2Cl_2]Cl$, a green-colored cobalt transition metal complex, which will be used to study spectrophotometry and kinetics in the experiments which follow.

The principal oxidation states of cobalt are the +2 and +3 states. The aqueous $[Co(H_2O)_6]^{3+}$ ion is very unstable because it is a powerful oxidizing agent and is readily reduced by water to $[Co(H_2O)_6]^{2+}$:

$$4[Co(H_2O)_6]^{3+} + 2H_2O \rightarrow 4[Co(H_2O)_6]^{2+} + O_2 + 4H^+$$

However, the +3 oxidation state is stabilized by replacing the coordinated water molecules with less labile ligands such as NH_3, NO, CN^-, and $NH_2CH_2CH_2NH_2$(en). A labile complex exchanges its ligands rapidly. Lability is associated with the d-electron configuration of the central metal. In general, ligands coordinated to a Co^{3+} ion do not dissociate from the Co^{3+} ion rapidly, and as a consequence, they are not easily replaced by other ligands. Thus, Co^{3+} complexes can endure many laboratory manipulations, and as we shall see, some of them can be prepared in structural forms whose stable existence depends on the durability of the bonds to the cobalt atoms.



Trans isomer Cis isomer

Figure 1. The *trans-* and *cis-* isomers of $[Co(en)_2Cl_2]Cl$.

In this assignment, you will prepare a complex ion with a net +1 charge in which two molecules of ethylenediamine (en) and two chloride ions are bonded to a central Co^{3+} ion. Each of the two

ethylenediamine molecules is attached to the Co^{3+} ion via the lone pairs on the basic nitrogen atoms at each end of ethylenediamine. Thus, six atoms (four nitrogen atoms and two chlorine atoms) are directly bonded to the cobalt atom and form the corners of an imaginary octahedron (eight-faced solid) with the cobalt atom at the center (see Figure 1). Notice that there are two possible structures for the complex.

In one structure (the *cis*- isomer), the two chlorine atoms occupy adjacent corners of the octahedron. In the other structure (the *trans*- isomer), the two chlorine atoms occupy opposite corners of the octahedron. These structurally different complexes have different physical and chemical properties. For example, the *cis* complex is dark purple in color, while the *trans* complex looks green.

In this experiment, you will prepare the chloride salt of the *trans* complex. The *trans* complex you prepare here will be used later in spectrophotometry and kinetics studies of its acid hydrolysis. Hereafter, we use the symbol "en" for the ethylenediamine molecule/ligand. It is very crucial that you measure all reagents and carry out all procedures exactly as described. Otherwise, you will synthesize an unintended complex or obtain a mixture of products.

Please read the appropriate chapter in your textbook as pre-laboratory preparation for this experiment.

Making Anti-Cancer Drugs

Cisplatin Transplatin

Geometric isomerism plays an important role in medicinal chemistry. For example, the complex on the left is know as "Cisplatin". It is one of the first sucessful anti-cancer drugs, and works especially well on tumors in reproductive organs. The trans isomer (right picture) has the identical molecular formula but does not exhibit any activity against tumors. It's amazing what a huge difference geometric isomerism can make!

Learning Goals

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

Laboratory	• Synthesizing metal coordination complexes • Isolation via crystallization • Vacuum filtration
Conceptual	• Crystal field theory • Solvation effects on crystal field and color • Geometric isomerism in transitional metal complexes
Data Analysis	• Balancing chemical reaction equations • Determining theoretical and experimental yield

Procedure

You will **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

You will need to retain the synthesized product for use in the Spectrophotometry and Kinetics experiments that follow.

Safety First

Take special caution when using ethylenediamine, 30% & 10% hydrogen peroxide, and 12 M hydrochloric acid; these three chemicals can cause severe burns.

Never attempt to smell any solution, as the odors can irritate mucous membranes in high concentrations.

Wear your goggles.

Stock Chemicals Used

Chemical	Maximum Amount Used
1.25 M Cobalt Chloride Solution	8 mL
3 M Ethylenediamine	8 mL
30% Hydrogen Peroxide	< 5 mL
95% Ethanol	12 mL
12 M Hydrochloric Acid	8 mL

Synthesis of *trans*-$[Co(en)_2Cl_2]Cl$

Safety First

Always keep the stock ethylenediamine bottle in the fume hood.

Don't take your flask containing ethylenediamine out of the fume hood until the reaction is complete.

1. Using a graduated cylinder, accurately measure out 8 mL of the $CoCl_2$ solution and then transfer this solution to a 50 mL Erlenmeyer flask. **In the fume hood**, add 8 mL of the ligand ethylenediamine ($C_2N_2H_8$) to the cobalt solution in the Erlenmeyer flask. Carefully make observations regarding any color or temperature changes and record these in your notebook.

Question A: Explain why the color changes occur during this reaction.

Question B: Write a balanced chemical equation for this process. The product is $Co(en)_2Cl_2$.

2. Return to the laboratory bench with your cobalt solution and make 6 mL of a 10% H_2O_2 solution from a 30% H_2O_2 stock solution. **Slowly** add the 6 mL of 10% H_2O_2 to the cobalt solution. Let the flask sit for 5 minutes at room temperature with occasional swirling and note any color or temperature changes. Transfer the solution to your small casserole. Rinse the flask with 5 mL of deionized water and add this rinse solution to the solution in the casserole.

Question C: Explain why the color changes during this process.

Question D: An excess of H_2O_2 has been used in this step since cobalt(II) is oxidized in the presence of the decomposing hydrogen peroxide. In the reduction half reaction of H_2O_2 in a basic solution hydroxide ions are formed, $2e^- + H_2O_{2(aq)} \rightarrow 2OH_{(aq)}$. Write a balanced ionic equation for the redox reaction occurring in step 2.

As a side note: The decomposition of hydrogen peroxide takes place as self-reaction involving both oxidation and reduction processes. Therefore, a by-product reaction occurring here is $2H_2O_{2(aq)} \rightarrow 2H_2O_{(l)} + 2O_{2(g)}$. This reaction is catalyzed by the presence of transition metal ions.

3. Place the small casserole over the steam bath in the fume hood. Be careful to avoid breathing the vapors that rise out of the casserole.

Safety First

Do not lean over and breathe the vapors from casserole.

Next, quickly but carefully, add 8 mL of the concentrated 12 M HCl to the casserole and note any color changes. Continue heating the casserole on top of the steam bath for about 60–70 minutes. During the 60–70 minutes, the volume of the cobalt solution should be reduced to one quarter of the original volume (you may want to use another casserole with the targeted volume of water for comparison).

As the volume of solution in the casserole is reduced, a crust of crystals (like a thin slurry) will begin to form on the surface of the liquid. Do not allow the solution to completely evaporate from the casserole. Thus, you may have to add a few mL of deionized water to avoid this, but only add a minimum. You may also need to add small amounts of water to the beaker in order to maintain this volume of water while boiling. As you the solution finishes heating, prepare an ice bath in your 400 mL beaker.

Question E: Write a balanced chemical equation for this process. The product is $[Co(en)_2Cl_2]Cl$.

4. Carefully remove the casserole from the steam bath and place it in an ice bath. Let the casserole cool for 5 minutes and then add 4 mL of 95% ethanol to force more of the water soluble product to crystallize. It is important here that you **allow the casserole to cool before rinsing** your product with the 95% ethanol. Allow 5 more minutes for the product to fully crystallize.

5. While the crystals are forming in step 4, set up the vacuum filtration apparatus as shown in Figure 2. This apparatus consists of a 125 mL filter flask, a black #3 neoprene adapter, and a plastic filtering funnel with a white fritted glass filter inside. Clamp the filter flask securely to the support rods. Wet the tip of the thick rubber tubing and connect it to the flask from vacuum supply in the lab.

Lab Skill Tips

The filter paper is very thin. Be sure you only have one sheet of filter paper.

Obtain a small circle of filter paper from the front of the room. Always use filter paper when filtering through a funnel. Place the filter paper against the fritted glass inside the funnel. Begin by turning on the vacuum. Seat the filter paper to the fritted glass by squirting a little deionized water on the filter paper. If everything is working properly, the paper should be pulled down against the fritted glass and the small spray of deionized water should be pulled through into the flask.

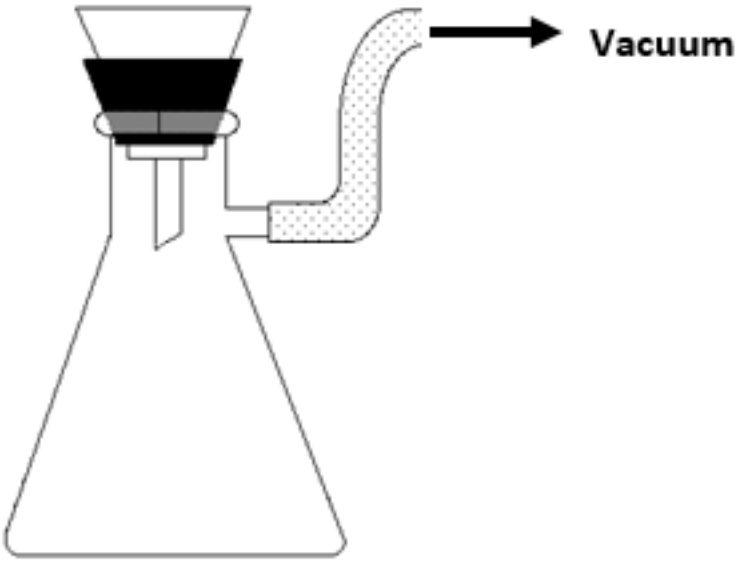


Figure 2: Filtering Apparatus

Hint

To avoid clogging the filter, first carefully pour the liquid into the funnel. Then, use a rubber policeman to transfer the solid.

6. Once the product has crystallized, remove the casserole from the ice bath and, using a rubber policeman, carefully transfer both the solid and the solution to the funnel. After the aqueous solution has been pulled through the filter, rinse the crystals twice with 4 mL portions of 95% ethanol while continuing to pull vacuum. You may use the ethanol to help transfer any product that remains in the casserole into the funnel. **Do not use water since the product is water soluble!**

   Continue to pull air through the crystals to dry the crystalline product. To collect the crystalline product, turn off the vacuum and then carefully disconnect the filtration flask from the vacuum line. Both you and your partner should do this together, with one person holding the filtration flask and the other person disconnecting the vacuum line; this will prevent your product and the solution in the flask from being spilled. The solution remaining in the Erlenmeyer flask must be disposed of in the metal ion waste container.

7. Clean an evaporating dish. Carefully transfer the damp product onto the dish. Place the evaporating dish over the boiling water bath for 5 minutes to finish the drying.

8. Weigh a clean, dry plastic snap-cap vial. Transfer the dry product to the vial and weigh. Calculate the mass of the product and the percent yield. Label the vial indicating molecular formula, the mass, the date, and the names of both you and your partner.

   Hand in your labeled vial to your TA before you leave. Your TA will retain your product for use in the Spectrophotometry and Kinetics experiments that follow.

Clean Up

- Remove the used circle of filter paper from your funnel and dispose of it in a crock pot.
- Rinse the filter flask, filter, and neoprene adapter thoroughly with deionized water and return these to the front of the room. Do not place this community equipment in your locker.

Safety First

Don't unplug or put back the hot plates while the top is still hot—you can tell the top is cool when the Caution light turns off.

Data Analysis

The following series of questions involve the chemistry in the synthesis of *trans*-$[Co(en)_2Cl_2]Cl$.

1. What can explain the color changes that take place when ethylenediamine ($C_2N_2H_8$) is added to the solution of cobalt(II) chloride?

2. Write the balanced chemical equation that represents the chemical reaction that takes place when ethylenediamine ($C_2N_2H_8$) is added to the solution of cobalt(II) chloride to form dichlorobis(ethylenediamine)cobalt(II)?

3. What can explain the color changes that take place when 6 mL of the 10% H_2O_2 is added to the solution of dichlorobis(ethylenediamine)cobalt(II)?

4. An excess of H_2O_2 has been used in this experiment since cobalt(II) is oxidized in the presence of the decomposing hydrogen peroxide. In the reduction half reaction of H_2O_2 in a basic solution, hydroxide ions are formed, $2e^- + H_2O_{2(aq)} \rightarrow 2OH^-_{(aq)}$. Write the balanced ionic equation for the redox reaction occurring when the hydrogen peroxide is added to dichlorobis(ethylenediamine)cobalt(II) solution.

5. Write the balanced equation for the reaction that takes place when concentrated hydrochloric acid is added to dichlorobis(ethylenediamine)cobalt(III) hydroxide solution.

   The following questions will lead you through the calculation of the percent yield of product.

6. What volume of the 1.25 M $Co[H_2O]_6Cl_2$ starting solution did you use?

7. What is the molar mass of $Co[H_2O]_6Cl_2$?

8. How many moles of $Co[H_2O]_6Cl_2$ were available in the starting material that you used?

9. The theoretical yield (in grams) of product is obtained by assuming that every mole of $Co[H_2O]_6Cl_2$ available as the starting material is converted to product *trans*-$[Co(en)_2Cl_2]Cl_2$. What value **in grams** is your theoretical yield of product?

10. What is the mass **in grams** of product, *trans*-$[Co(en)_2Cl_2]Cl$, that you collected? Your mass precision should be to a thousandth of a gram.

11. Based on your calculated theoretical yield and your reported mass of product, calculate the percent yield of product, *trans*-$[Co(en)_2Cl_2]Cl$? Report your percent yield to a tenth of a percent, i.e. 45.3%.

Conclusion.

Briefly discuss interpretations of your observations and results. Include in your discussion, any conclusions drawn from the results and any sources of error in the experiment.

A Spectroscopy Study

Spectrophotometric Analysis of trans-$Co[(en)_2Cl_2]Cl$ and the Aquation Product, $[Co(en)_2(H_2O)Cl]Cl_2$

Introduction

In the spectrophotometric portion of this experiment you will analyze *trans*-$[Co(en)_2Cl_2]Cl$ that you previously prepared and its acid catalyzed hydrolysis product, $[Co(en)_2(H_2O)Cl]Cl_2$. You will compare the visible absorption spectra of the two cobalt complexes in aqueous solution, examine how their separate absorptions depend on the concentrations of the cobalt complex solutions, and use the combined absorptions to determine the concentrations of the two cobalt complexes in an aqueous mixture.

A Brief Introduction to Spectrophotometry

The use of spectral data to identify and quantify substances is fundamental to chemical analysis. You are already familiar with the absorption spectra that are produced when atoms of an element absorb photons of electromagnetic radiation of certain wavelengths and cause an electronic transition from lower to higher energy states. You learned in Chemistry 2A that each element has a characteristic absorption spectrum.

Many substances absorb photons in the visible light region of electromagnetic radiation spectrum while reflecting other photons in the visible light region. As a result of the reflected photons, a particular substance appears a certain color. Visible light, therefore, may be used to study colored substances. This is referred to as spectrophotometry. For example, a blade of grass is green, because its chlorophyll absorbs red and blue wavelengths strongly and green less strongly, so most of the green is reflected, as shown by Figure 1 below.

The height and shape of the curve make up a characteristic absorption spectrum for a substance and can be used for identification purposes. The curve varies in height because chlorophyll absorbs incoming wavelengths to different extents. The absorption spectra is of chlorophyll *a* and chlorophyll *b* is shown below. There is high absorption of red and blue frequency photons, hυ.

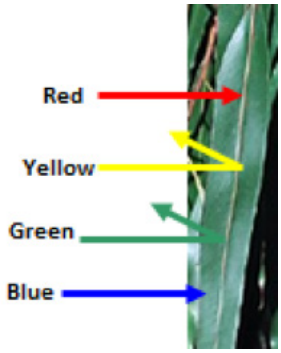


Figure 1. The absorption spectra of chlorophyll *a* and chlorophyll *b*.

A spectrophotometer (or spectrometer, for short) is used to measure absorption spectra and is a combination of a light source that emits a continuous band of radiation, a monochromator (usually a reflection grating with associated optics) to select a narrow range of wavelengths, and a photoelectric detector to measure the light intensity, as indicated in Figure 2.

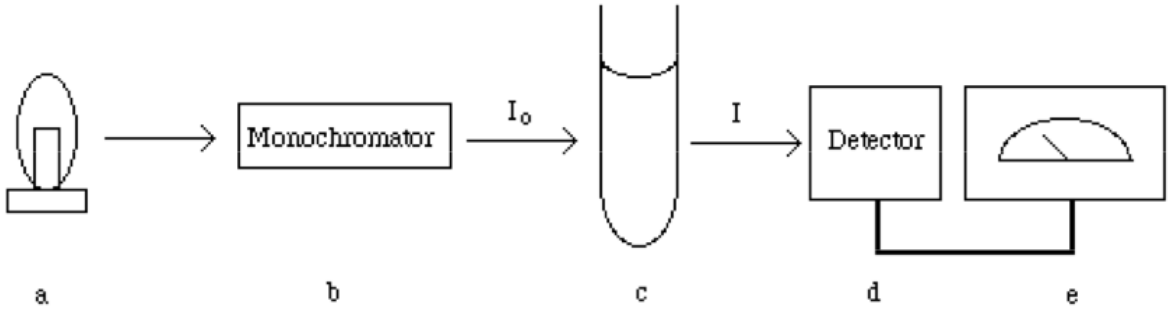


Figure 2: Schematic Representation of a Spectrophotometer.

In addition to identifying a substance, an absorption spectrum can be used to determine its concentration because the absorbance, the amount of light of a given wavelength absorbed by a substance, is proportional to the number of molecules present in the sample. This relationship is called the Beer-Lambert Law or more simply Beer's Law. Consider monochromatic light of a given intensity incident on a sample, as shown in Figure 2. If this light can be absorbed by the sample, then the transmitted light will have a lower intensity than the incident light. The transmittance, T, is defined as the ratio of the transmitted intensity, I, to the incident intensity, I_0,

$$T = \frac{I}{I_0}$$

The percent, %T, is simply 100 · T. The transmittance is decreased if either the concentration, C, of absorbing substance is increased or the path length, ℓ, of the sample is increased, since both increase the number of absorbing species in the path of the light.

A related quantity is the absorbance, A, which is given by

$$A = -\log T = -\log_{10}\frac{\%T}{100} = 2 - \log_{10}(\%T)$$

%T is read directly from the spectrometer. However, the absorbance is particularly important since it, and not the transmittance, is directly proportional to the concentration of the absorbing substance and the path length. This proportionality constitutes Beer's Law, and is commonly written as

$$A = \varepsilon \ell C.$$

The concentration, C, is expressed in moles per liter (M) and the path length, ℓ, is generally expressed in cm. The quantity ε is called the molar absorptivity and has units of $M^{-1}\,cm^{-1}$. Note that the quantity εℓC is dimensionless, as are both the absorbance, A, and the transmittance, T. The molar absorptivity is characteristic of the substance. It tells us how much

light the substance absorbs at a particular wavelength. A graphical plot of either the absorbance at constant path length or the molar absorptivity versus wavelength is called the absorption spectrum of the substance.

Beer's Law forms the basis for the analytical use of spectroscopy to determine concentrations. As indicated by the equation of Beer's Law shown above, a plot of the absorbance at a given wavelength for a particular species versus the concentration of the species yields a straight line with a slope equal to $\varepsilon\ell$ and an intercept of zero. Since the path length of the cell used for the absorbance measurements is typically known, the molar absorptivity of the species at a chosen wavelength is readily determined from such a Beer's Law plot. The concentration of the species in an unknown sample can then be determined by measuring the absorbance of the sample at the same wavelength in any cell of known path length.

The wavelength for such an analysis should be chosen so that small changes in wavelength do not yield large changes in absorbance. Namely, the chosen wavelength should be in a relatively flat portion of the absorption spectrum. Typically, a wavelength associated with a maximum in the spectrum is chosen, since at a maximum, the slope of the spectrum is zero or horizontal and simultaneously good sensitivity is obtained in the analysis (significant absorbance for a given concentration).

One should always establish, before its analytical use, that Beer's Law is followed by a species over the concentration range of interest, since deviations from Beer's Law often occur at high concentrations. Typically, these deviations can be traced to changes in the absorbing species or the bulk solution with concentration. For example, in concentrated solutions the solute molecules are closer together on average and interact with each other, changing their energy levels and spectroscopic properties from a dilute solution. The species of interest also may exist in equilibrium with other species that have different molar absorptivities. In such cases, a graph of absorbance versus concentration will appear to deviate from a straight line at high concentrations.

If two species are present, and neither affects the light absorbing properties of the other, then the observed total absorbance is simply the sum of the absorbances of the individual species. When this is true, the individual concentrations can be determined from spectrophotometric measurements. Because interactions often do arise, sometimes when least expected, the absorption spectra of the species should be investigated when they are separate and when they are simultaneously present to determine whether the absorbances are indeed additive before any analytical spectrophotometric measurements.

Swimming Pool Upkeep

To ensure a refreshing and safe swimming experience, a well-maintained pool is essential. This means not just clean water but also a balanced water chemistry. Continual maintenance requires regular monitoring of the pool's pH, water hardness (mineral concentrations), alkalinity (concentration of carbonate and bicarbonate anions), and chlorine concentration to keep the water in optimal condition. Ideally, chlorine levels should range between 1 ppm to 4 ppm, as levels lower than this can expose swimmers to bacterial contamination, while higher levels may cause skin irritation.

Although test strips are commonly used to check chlorine concentration in pool water, advanced monitoring equipment can be used for greater accuracy. One such instrument is a spectrophotometer, which employs a beam of light to measure the absorption of light by various molecules in a sample. This method is based on Beer's Law, which states that the amount of absorption of a substance is directly proportional to its concentration in the sample. By comparing mesured data with known standards, the spectrophotometer provides an accurate measurement of chlorine concentration in pool water.

Spectroscopy is a more accurate method of measuring chlorine concentration enabling pool owners to adjust the chlorine levels in their pools for a safer and more enjoyable swimming environment.

Learning Goals

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

Laboratory	• Dilutions • Calibrating spectrometer • Accurately finding lambda max
Conceptual	• Beer's Law • Fundamentals of spectroscopy • Electromagnetic spectrum • Absorbance vs. concentration
Data Analysis	• Beer's Law plots • Solving for epsilon (ε) • Solving for an unknown concentration

"Determining the Blank Cuvette" on page 58

Operation Of Genesys™ 20 Spectrophotometer

Spectrophotometers all contain some kind of light source (visible instruments often use just a tungsten bulb); various mechanical and optical elements which are used to create a directed and collimated beam; a wavelength selector of some sort (often either a diffraction grating or a prism); an enclosed cell compartment to hold the absorbing sample; a radiation detector (often a photocell or more commonly now a photodiode); and some kind of readout device (e.g. a needle deflection on a meter).

It is important that you understand the operation of the GENESYS™ 20 before you begin this laboratory. The GENESYS™ 20 is an expensive and sensitive instrument and must be operated carefully and intelligently.

The light source in a GENESYS™ 20 is an ordinary tungsten lamp whose radiation extends over the entire visible range. The light from the lamp passes through an entrance slit and is dispersed by a diffraction grating. The grating can be rotated so that a small band of selected wavelengths from the dispersed beam passes through an exit slit, and then through the cell (cuvette) containing the sample. The cuvettes used with a GENESYS™ 20 have a path length (internal diameter) of 1.00 cm. The light transmitted through the sample strikes a solid-state silicon detector that generates an electrical signal proportional to the radiant power (light intensity). The signal from the detector drives a meter that can be calibrated to read transmittance or absorbance.

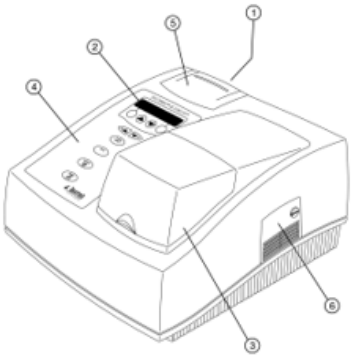
The *calibration procedure* entails setting 0 Absorbance at a given wavelength with a cuvette containing a reference or blank solution. The blank solution is missing the component of interest, but is otherwise as identical as possible to the solution to be analyzed for the component of interest.

Typically, the blank solution is just the solvent. This is required since the output of the lamp and the sensitivity of the detector varies with wavelength. The electronics of the instrument automatically sets 100% absorbance. An identical cuvette containing the solution of interest is then inserted into the spectrometer, and the absorbance is read from a meter on the instrument.

Both the calibration and the reading must be done at the same wavelength. The reading for the solution then represents the absorbance at the chosen wavelength due to the component of interest. The calibration has accounted for any absorption (or reflection or scattering) of light by the cuvette and other species in the reference solution.

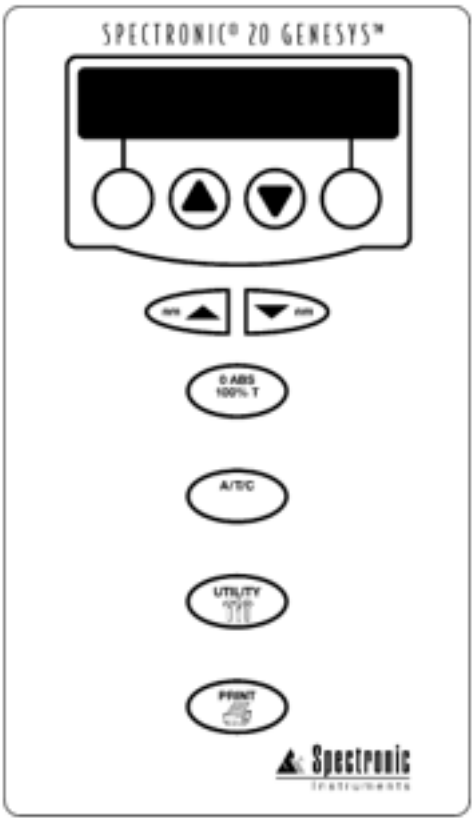
GENESYS™ 20 Spectrophotometer

1. On / Off switch
2. LCD display
3. Sample compartment hood
4. Keyboard
5. ----
6. Lamp compartment door



The power switch is located on the bottom left in the back of the instrument. When you turn on your GENESYS™ 20 spectrophotometer, it performs its power-on sequence. This sequence includes checking the software revision, initializing the filter wheel and the monochromator. The power-up sequence takes about two minutes to complete. Allow the instrument to warm up for 10 minutes before using it.

Keyboard Layout of GENESYS™ 20 Spectrophotometer



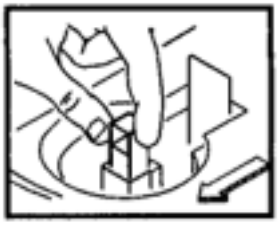
Absorbance and %Transmittance Measurements

Be sure the cell holder is empty before turning on the instrument.

1. Press **A/T/C** to select the absorbance or % transmittance mode. The current mode appears on the display.

2. Press **nm** ⋏ or **nm** ⋎ to select the wavelength.

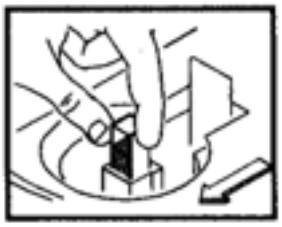
Note: Holding either key will cause the wavelength to change more quickly.



3. Insert your blank into the cell holder and close the sample door.

Note: Position the cell so the light (indicated by the arrow in drawing) passes through the clear walls.

4. Press **0 ABS/100%T** to set the blank to 0 A or 100% T.



5. Remove your blank and insert your sample into the cell holder. The sample measurement appears on the LCD display.

Error Messages

This section lists the messages generated to alert you of errors or other abnormal conditions. The instrument recognizes two types of errors. With the first type, the instrument is still functional; with the second, the instrument is not functional until the condition is resolved.

1. Flashing data display: The sample has an absorbance or a transmittance value below or above the photometric range of the instrument. The display flashes until the condition is resolved.

2. Sample too dark: The instrument has been asked to zero a sample with a high absorbance at a low energy point. The instrument beeps three times to announce the message, the message remains on the display for two seconds, then the normal display returns.

3. Sample too bright: The instrument has been asked to zero a sample while the door of the sample compartment is open. The instrument beeps three times to announce the message, the message remains on the display for two seconds, then the normal display returns.

Determining the Blank Cuvette

1. Obtain a pair of cuvettes. One will be used for the blank solution and the other will be used for the standard and unknown samples.

2. Fill both cuvettes with the solvent and wipe the outside with a Kimwipe to make sure it is clean and dry (no fingerprints). Be sure to always add enough solution to reach to within 1/8 of the triangular mark on the cuvette. This will insure that all incident radiation passes through the solution. It is also critical that the **triangular** mark on the cuvette be facing you when inserted in the sample holder for all measurements.

3. Insert one filled cuvette into the sample holder that is located under the hood to the left. Close the hood.

4. Calibrate to 0 absorbance by pressing the 0 ABS/100% T key. Remove the cuvette, insert the other cuvette, and read the absorbance for this second cuvette. If the reading is less than 0 absorbance, then this second cuvette will be used as the blank cuvette; if the reading is greater than 0 absorbance, then the first cuvette will be used as the blank cuvette.

Calibrating the Instrument

The calibration procedure must be performed for absorbance measurements taken at each different wavelength.

1. Select the desired wavelength by pressing the up or down "wavelength selector" arrow keys of the key pad of the instrument.
2. Insert the "blank" cuvette containing the blank reference solution (which is acidic solvent for this experiment) into the sample holder and close the hood.
3. Calibrate to 0 absorbance by pressing the 0 ABS/100%T key.
4. Remove the "blank" cuvette. **Do not re-adjust any settings at this point.** You are now ready to insert the other cuvette containing the sample of choice for a measurement at this particular wavelength.
5. Repeat steps 1–4 for all measurements that take place at different wavelengths.

Question A: Why do we allow electronic instruments to warm-up before use?

Question B: Why is it important not to have fingerprints on the cuvette?

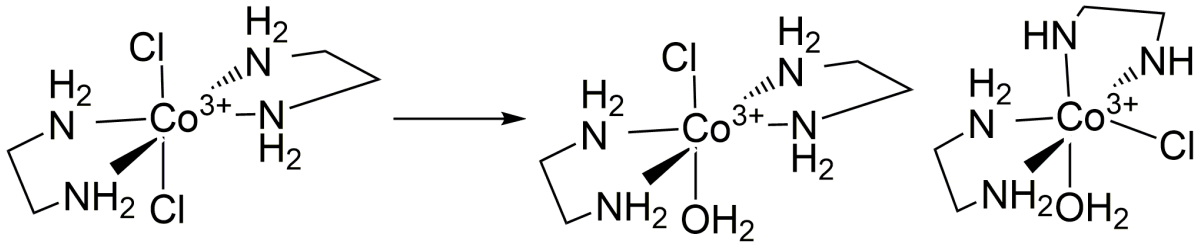
Question C: Why is it important to align the cuvette in the sample holder the same way each time?

The Experiment

In the present exercise you will experimentally determine the wavelengths of the visible absorption maxima of two non-interacting compounds and measure the molar absorptivity for both of them at these two different wavelengths. Then, you will measure the absorption of an unknown mixture of two compounds in order to determine the concentration of each species.

The two compounds referred to above are the green "chloro" complex synthesized in the last experiment and a red "aquo" derivative, which you will make from the solution of the green complex.

In acidic solutions with low free chloride ion concentrations, one of the chloride ions in the *trans*-$[Co(en)_2Cl_2]^+$ complex ion can be replaced by a water molecule to give an equilibrium mixture of the *cis* and *trans* isomers of the aquo complex, $[Co(en)_2(H_2O)Cl]^{2+}$. This reaction is accompanied by a color change from green to red:



$$trans\text{-}[Co(en)_2Cl_2]Cl + H_2O \xrightarrow{H^+} cis\text{-}\,\&\,trans\text{-}[Co(en)_2(H_2O)Cl]Cl_2$$

"chloro" complex (green color) **"aquo" complex (red color)**

Throughout this experiment, the green-colored *trans*-$[Co(en)_2Cl_2]Cl$ will be referred to as the **chloro** complex, while the red-colored mixture of the *cis* and *trans* isomers of the hydrolysis product, $[Co(en)_2(H_2O)Cl]Cl_2$, will be simply referred to as the **aquo complex**.

In the first part of this experiment you will determine the wavelength of maximum absorbance (λ_{max}) for the two complexes. Then, you will prepare four solutions of each complex of known concentrations. You will then measure the absorbance of each solution using a GENESYS™ 20 spectrophotometer at the wavelength corresponding to the maximum absorbance for the chloro complex and will graph the data, with concentration on the x-axis and absorbance on the y-axis, to form a Beer's Law plot. By finding the slope and knowing the path length of your cuvette cell, you will be able to calculate the molar absorptivity coefficient at that wavelength for that complex. You will repeat this for the second wavelength that corresponds to the maximum absorbance position for the aquo complex. These measurements (i.e. at both wavelengths) will be carried out for solutions of chloro and aquo complexes. In the final portion of this experiment, you will analyze solutions that contain a mixture of the two complexes. You will determine the concentrations of both complexes in the mixture using your spectrophotometric measurements.

"Determining the Blank Cuvette" on page 58

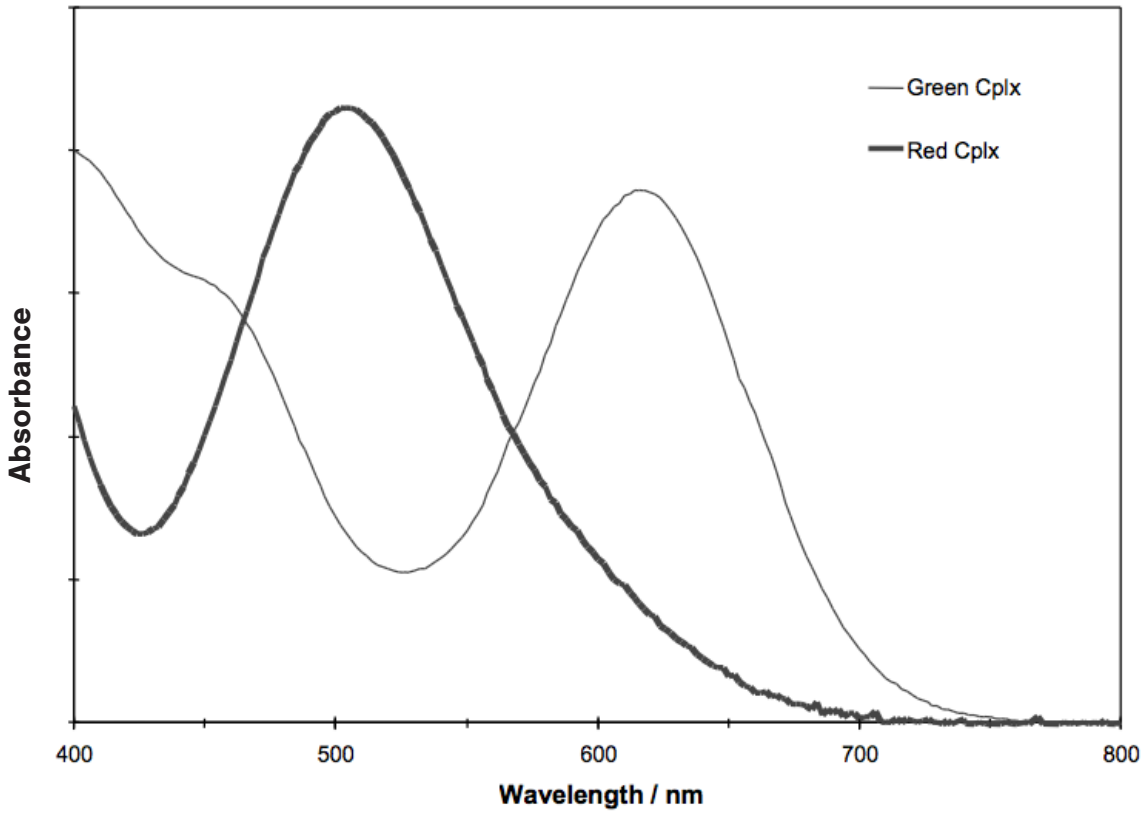


Figure 4. Absorption Spectra of Cobalt(III) Ethylenediamine Complexes

Procedure

You will **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
3 M Sulfuric Acid	12 mL
Trans-$[Co(en)_2Cl_2]Cl$	0.2 grams

Safety First

Treat the spectrophotometers with great care as they are expensive and delicate instruments.

Wear your gloves and goggles at all times.

Lab Skill Tips

Clamp the flask securely or use a small enough beaker to prevent it from overturning in the ice water bath.

Part I. Solution Preparation and Aquation of the Chloro Complex.

Prepare 120 mL of 0.3 M sulfuric acid to be used as a solvent for the next two experiments.

1. Use a 25 mL buret to dispense a measured 24 ± 0.2 mL of this solvent into a clean 50 mL Erlenmeyer flask. Place this 50 mL flask to above the liquid level in a 250 mL beaker containing an ice bath made roughly of equal parts of water and ice.

2. Accurately weigh ca. 200 ± 4 mg sample of the green chloro complex and transfer it carefully to the flask of cooled, acidic solvent. Remove the flask from the cooling bath, swirl it several times to ensure that the solid has completely dissolved and the solution is homogeneous, and then quickly fill another a 25 mL buret with this cold solution. Return the flask containing the rest of the green solution to the ice bath.

3. Dispense a measured 12.0 ± 0.2 mL portion of the cold green solution into another clean Erlenmeyer flask and then quickly return the green solution remaining in the buret to the still-cooling flask from which it originally came. At this point you should have two essentially identical 12.0 mL samples of the green solution in two different flasks—one of them in an ice bath and the other one out on the bench top warming to room temperature.

4. Put the "warmer" flask into a beaker that contains boiling water for about 10 minutes, which should cause the green starting material to be converted entirely to the red aquo product. Cool the flask containing the red solution to room temperature. If you wish you can use an ice bath to speed up the cooling.

Part II. Spectral Assignments.

Figure 4 contains the absorption spectra of the red and green complexes. You will need to determine the true absorption maxima of the two complexes. The instrument you will use to make this determination is the manually operated GENESYS™ 20 spectrophotometer.

1. You should start with the red solution. The red solution is stable so it is clearly the one to use to familiarize yourself with the instrument and experimental procedure.

2. Your red stock solution is a little too concentrated to give good results. Therefore, you will want to use a disposable pipet to put about 1 mL of it in a test tube and then dilute that with about 3 mL of the acidic solvent dispensed from the 25 mL buret.

3. Obtain a pair of cuvettes. One will be used for the "blank" solution and the other will be used for the standard and unknown samples. Determine which cuvette will be used for the "blank" solution by following the procedures given on the section "Determining the Blank Cuvette" on page 60.

4. Fill the cuvette selected for the blank to within ⅛" of the triangular mark on the cuvette with the acidic solvent dispensed from the 25 mL buret.

5. Rinse the other cuvette with a small portion of the diluted red solution (aquo product) and then fill it to within ⅛" of the triangular mark on the cuvette with this solution.

6. From the scanned spectra given in Figure 4 shown above, estimate the wavelength of the absorption maximum of the red colored complex.

7. Select a wavelength that is about 10 nm lower than the estimated absorption maximum determined in step 6. Calibrate the instrument following the procedure given on page 58, under the section, "Calibrating the Instrument." Once you have established 0 absorbance using your blank solution, insert the sample cuvette and read the absorbance. Record your measurement.

8. Remove the sample cuvette, increase the wavelength by two nanometers (nm), and recalibrate the instrument using the blank cuvette. Re-insert the sample cuvette and read the absorbance. Record your measurement.

9. Repeat step 8 until you have taken absorbance measurements every two nanometers from 10 nm below your estimate to 10 nm above your estimate. This can be done fairly quickly in pairs, with one person recalibrating, measuring, and calling out the resulting absorbance values and the other one recording the data. Switch roles half way through the process so each partner has an opportunity to perform both tasks.

10. Repeat steps 2 and 5–10 above for the green solution (chloro complex) instead of the red and thereby find the true wavelength of the green complex absorption maximum. You need to work efficiently because at room temperature the green solution changes slowly with time. For step 2, mix 1 mL of the cold stock solution with 3 mL of solvent at room temperature and use the resulting solution immediately unless water condenses on the outside of the test tube in which you did the mixing. If that happens, hold the test tube tightly in your hand until it warms up to above the dew point. The absorbance measurement will be greatly affected if condensation occurs on the outside of the cuvette. For step 7, be sure to use the estimated wavelength of maximum absorption for the green solution.

11. Review your absorbance readings for the two complexes. Determine the wavelength where maximum absorbance occurred for each colored complex. Record these wavelengths for use in Part III.

Green Chem

Reduce waste by reusing the same cuvette throughout the lab.

Empty the sample cuvette. Rinse it with deionized water, and carefully blot it dry using a Kimwipe.

Part III. Collecting the Data Needed to Construct Beer's Law Plots.

In this part of the experiment, you will prepare eight solutions with known concentrations—four each of both the red and green solutes—and measure their absorbances at the wavelengths of the two absorption maxima you determined in Part II above; i.e., you will make a total of sixteen spectroscopic measurements.

1. Fill one 25 mL buret with the cold, green chloro solution and another one with the solution of its red aquation product. Evenly splitting up the workload within the partnership, dispense four measured 2.0 ± 0.2 mL portions of each of these solutions into separate clean test tubes. This entails numerous buret readings, but in order to maintain a reasonable degree of precision here you really do have to make them all. As always, immediately store the "green" tubes in the ice/water bath.

2. To the four "red" test tubes in turn use the 25 mL buret to add measured volumes of the acidic solvent as follows: 3.0 ± 0.2; 4.0 ± 0.2; 5.0 ± 0.2 and 6.0 ± 0.2 mL. (You should now have four tubes containing accurately measured total volumes of about 5, 6, 7, and 8 mL.)

3. With the proper technique measure the absorbance of each of these solutions at the wavelengths you determined above for the absorption maxima of both the red and the green complexes (for a total of 8 readings.)

4. One test tube at a time, repeat steps 2 and 3 with the four samples of the green complex. As before, you can use the diluted samples immediately unless water vapor condenses on the outside of the test tube after mixing—in which case, warm it up with your hand.

5. When you are finished with Part III, you should have collected a total of 16 different absorbance readings here; 8 for the red solutions and 8 for the green solutions.

Part IV. Collecting Data for the Analysis of a Mixture of the Two Complexes.

Your TA will give you a solution that contains a mixture of the red and green complexes prepared by allowing a solution of the chloro complex to partially convert to the aquo product.

Measure the absorbance of the mixture at the wavelength of maximum absorbance found for green solution in Part I and measure the absorbance of the mixture at the wavelength of maximum absorbance found for the red solution in Part I. (You should have a total of 2 readings.)

Clean-up

- Dispose the solutions from test tubes into Cation Metal Waste.
- Neutralize unneeded sulfuric acid with **0.5 g sodium bicarbonate** before sink disposal.
- Store the 0.3 M sulfuric acid in a 1L bottle for use next week.

Data Analysis

Part III. Making the Beer's Law Plots.

Overview and a Modest Theoretical Excursion: In this part of the experiment you will manipulate the data collected in Part III to obtain the values of effective molar absorptivity, symbolized by ε*, for both the red and the green complexes at the two wavelengths of interest here.

Beer's Law in the form in which you have seen it previously, $A = \varepsilon \ell C$, strictly applies only to a slender beam of monochromatic light passing through a cell with flat, parallel windows. In the present experiment the light beam is not small and the cell is not a rectangular parallelepiped, so the path length, ℓ, cannot be defined.

Operationally, the detector outputs a signal that is the average of the exit intensity over the cross section of the cylindrical cuvette. If necessary one could measure what might be called an "effective path length" for each cuvette by measuring the absorbance of a solution of some absorber whose true molar absorptivity has already been measured, but we do not have to do this here.

Instead, we can combine the molecular property ε and the effective path length of the cell being used for all the measurements into a new constant, ε*, whose magnitude is valid only for the particular system (absorber and cuvette) under study. Beer's Law then takes the simpler form $A = \varepsilon^* C$. (Note that the way it is defined here ε* must have dimensions M^{-1} because absorbance is dimensionless.)

We shall henceforth distinguish the four different ε* values by two-letter identifying subscripts: namely, GG, GR, RG and RR. The meanings of these combinations are as follows:

GG = Green solution examined at the wavelength of the Green absorption maximum

GR = Green solution examined at the wavelength of the Red absorption maximum

RG = Red solution examined at the wavelength of the Green absorption maximum

RR = Red solution examined at the wavelength of the Red absorption maximum

According to the modified form of Beer's Law introduced above, one can determine the numerical value of ε^* ($= \varepsilon\ell$) for some solute at a given wavelength by determining the slope of a plot of absorbance vs. concentration.

Microsoft Excel is **strongly recommended** for the following calculations:

1. Calculate the molar concentrations of the eight solutions you studied.
2. Create graphs by plotting absorbance vs concentration using the 16 absorbance readings you have collected, along with their corresponding concentrations. The graphs you need to prepare are:
    a. Green solution at the wavelength of green solution absorption maximum
    b. Green solution at the wavelength of red solution absorption maximum
    c. Red solution at the wavelength of green solution absorption maximum

d. Red solution at the wavelength of red solution absorption maximum

3. Determine the line of best fit for each set of points and find the four relevant values of ε* where the line of best fit passes through the point (0, 0).

Part IV. Spectrophotometric Analysis of a Mixture of the Two Complexes.

Theory: The absorbance of a mixture of absorbing species that do not interact with each other in any way is simply the sum of the absorbances of the individual species. This means that in a solution that contains both the red and the green complexes in significant amounts the absorbance at λ_{max} of the green form will be given by the equation

$$A^G = \varepsilon^*_{GG}[C_G] + \varepsilon^*_{RG}[C_R]$$

and the absorbance at λ_{max} of the red form will be given by

$$A^R = \varepsilon^*_{GR}[C_G] + \varepsilon^*_{RR}[C_R]$$

where $[C_x]$ is the molar concentration of X = R, G.

It follows from this that if the four ε* values are all known, the individual concentrations in a mixture of the two species can be obtained by measuring its absorbance at the two maximal wavelengths and solving the pair of simultaneous equations given just above. (Students who know some linear algebra will recognize that these equations can be compacted into the matrix equation.)

Calculate the concentrations of the green "chloro" complex and the red "aquo" complex in the mixture.

The following series of questions pertains to the analysis of the data in Parts III and IV. Your data and resulting calculations will be verified as you proceed through the exercise. You should have worked through the analysis of Parts III and IV in your notebook before beginning; otherwise, you may need an hour or so to complete this exercise. You may leave the exercise at any point and continue it later.

1. Why do we allow electronic instruments to warm-up before use?

2. Why is it important not to have fingerprints on the cuvette?

3. Why is it important to align the cuvette in the sample holder the same way each time?

4. In part I step 2, you were instructed to weigh approximately 200 mg of the green chloro complex. What is the precise mass **in milligrams** of the green chloro complex you used to prepare the solution?

5. In part I step 2, you were instructed to dissolve the green chloro complex into approximately 24 mL of acidic solvent. What is the precise volume in milliliters of the acidic solvent you used to prepare the solution (e.g. 24.31)? Your precision should be to a hundredth of a milliliter.

6. If you weighed out 203 mg of the green chloro complex and dissolved it in 24.14 mL of acidic solvent, the molarity of your stock solution would be 0.0295 M. Using your precise value of mass and volume that you entered above, calculate the concentration of the original green chloro complex stock solution in moles per liter.

7. What is the wavelength **in nanometers** of the maximum absorbance for the GREEN solution?

8. What is the wavelength **in nanometers** of the maximum absorbance for the RED solution?

9. The original red stock solution has the same concentration as the green stock solution since one mole of the red aquo complex forms for each mole of the green chloro complex that is converted. Therefore, the concentrations of the two sets of four diluted solutions, the red and green, are equal for each respective concentration.

   For each set of diluted solutions, calculate the values of the concentration of the solutions you prepared. Write them in descending order (highest to lowest) in units of moles per liter.

10. Next, you will need your data for the absorbance of each of your diluted samples for both the green and red solutions that is used subsequently to verify your Beer's law plots. We will refer to your Beer's law data using the shorthand suggested in your laboratory manual.

    GG, GR will be the measurements of the green solution at the maxima of the green and red complexes respectively and RG, RR will be the measurements on the red solution at the maxima of the green and red complexes respectively. It is important to correspond the absorbance readings with the correct diluted sample. This means that your absorbance values will be in decreasing order. Please check for this carefully as it will affect the slope calculations.

11. **Determination of the GG effective extinction coefficient.**

    a. Record the four values of the GG absorbance measurement in corresponding order of solution concentration (high concentration to low concentration; values of absorbance will be running in the same sense, high absorbance to low absorbance and expressed to three significant digits). The absorbance values should have a value between 0 and 1.

    b. Plot the values of GG Absorbance versus Concentration on a spreadsheet or a programmable calculator, and carefully determine the slope of the best straight line fit with intercept (0, 0) through the data.

    The slope values for GG, GR, RG, and RR must be accurately determined because they will enable you to solve for the unknown concentration of your mixture of the two cobalt complexes.

    c. What is the value of the slope of A vs. C. This is your effective extinction coefficient for GG?

12. **Determination of the GR effective extinction coefficient.**

    a. Record the four values of the GR absorbance measurement in corresponding order of solution concentration (high concentration to low concentration; values of absorbance will be running in the same sense, high absorbance to low absorbance and expressed to three significant digits). The absorbance values should have a value between 0 and 1.

    b. Plot the values of GR Absorbance versus concentration on a spreadsheet or a programmable calculator, and carefully determine the slope of the best straight line fit with intercept (0, 0) through the data.

    The slope values for GG, GR, RG, and RR must be accurately determined because they will enable you to solve for the unknown concentration of your mixture of the two cobalt complexes.

c. What is the value of the slope of A vs. C. This is your effective extinction coefficient for GR?

13. **Determination of the RG effective extinction coefficient.**

a. Record the four values of the RG absorbance measurement in corresponding order of solution concentration (high concentration to low concentration; values of absorbance will be running in the same sense, high absorbance to low absorbance and expressed to three significant digits). The absorbance values should have a value between 0 and 1.

b. Plot the values of RG Absorbance versus concentration on a spreadsheet or a programmable calculator, and carefully determine the slope of the best straight line fit with intercept (0, 0) through the data.

The slope values for GG, GR, RG, and RR must be accurately determined because they will enable you to solve for the unknown concentration of your mixture of the two cobalt complexes.

c. What is the value of the slope of A vs. C. This is your effective extinction coefficient for RG?

14. **Determination of the RR effective extinction coefficient.**

a. Record the four values of the RR absorbance measurement in corresponding order of solution concentration (high concentration to low concentration; values of absorbance will be running in the same sense, high absorbance to low absorbance and expressed to three significant digits). The absorbance values should have a value between 0 and 1.

b. Plot the values of RR Absorbance versus concentration on a spreadsheet or a programmable calculator, and carefully determine the slope of the best straight line fit with intercept (0, 0) through the data.

The slope values for GG, GR, RG, and RR must be accurately determined because they will enable you to solve for the unknown concentration of your mixture of the two cobalt complexes.

c. What is the value of the slope of A vs. C. This is your effective extinction coefficient for RR?

15. **You are now ready to complete the analysis of Part IV data.** Using the values of the effective extinction coefficients you determined from your slopes and the values of the absorption you obtained for the mixture at the spectral maxima for the green and red solutions, solve the 2x2 simultaneous linear equations for the concentrations of the green and red complexes in the mixture.

a. Record the absorbance you measured for the mixture of cobalt complexes at the wavelength of absorption maximum for the GREEN complex. The absorbance values should have a value between 0 and 1.

b. Record the absorbance you measured for the mixture of cobalt complexes at the wavelength of absorption maximum for the RED complex. The absorbance values should have a value between 0 and 1.

c. Calculate the concentrations of the green and red complexes in the mixture.

A Kinetics Study

Kinetics Studies of trans-Co[(en)$_2$Cl$_2$]Cl and the Aquation Product, [Co(en)$_2$(H$_2$O)Cl]Cl$_2$

Introduction

In this segment of the experiment, you will be studying kinetics based on the same acid catalyzed hydrolysis reaction of the green chloro complex you previously prepared. Recall, in acidic solutions with low free chloride ion concentrations, one of the chloride ions in the *trans*-$[Co(en)_2Cl_2]^+$ complex ion can be replaced by a water molecule to give an equilibrium mixture of the cis and trans isomers of the aquo complex, [Co(en)2(H2O)Cl]$^{2+}$. This reaction is accompanied by a color change from green to red:

$$trans\text{-}[Co(en)_2Cl_2]Cl + H_2O \xrightarrow{H^+} cis\text{-}\,\&\,trans\text{-}[Co(en)_2(H_2O)Cl]Cl_2$$

"chloro" complex (green color) **"aquo" complex (red color)**

You will take advantage of a color change in this reaction to determine both the order of reaction, and the activation energy required to initiate the hydrolysis.

In this segment of the experiment, you will be studying kinetics based on an acid catalyzed hydrolysis reaction of the green chloro complex you previously prepared. In particular, you will determine both the order of reaction, and the activation energy required to initiate the hydrolysis.

As you have previously studied, the rates of chemical reactions can usually be expressed as algebraic functions of the concentrations of the reacting species. In many cases, the rate of reaction is proportional to the product of the concentrations of the reactants, each raised to a power called the *order* of the reaction with respect to that reactant:

$$\text{Rate} = k[A]^a[B]^b$$

where the exponents *a* and *b* are referred to as the orders with respect to the reactants A and B; and *k* is the rate constant. In most cases, the orders, *a* and *b*, are zero, one, or two.

Read the appropriate chapter in your textbook as pre-laboratory preparation before beginning this experiment.

The first two parts of this exercise will be to determine the order for the hydrolysis reaction with respect to the $[Co(en)_2Cl_2]^+$ ion. For one of the two parts, you will be given data that was collected previously for the decrease in $[Co(en)_2Cl_2]^+$ ion over time, from which you can deduce the order of the reaction. Specifically, by finding the best function of chloro complex concentration that gives the best linear correlation, we can determine the order of the reaction.

For the other part, you will use a complementary method for determining the order of reaction. You can examine half-lives using the following rationale for Part I. At a particular acid concentration, you may assume that the hydrolysis rate can be expressed by the relation:

$$\text{Rate} = k[Co(en)_2Cl_2^+]^n$$

where $n = 0$, 1, or 2, and here the square brackets indicate molar concentrations. To find the order of this reaction we will measure the dependence of the initial concentration, C_0, of the $[Co(en)_2Cl_2]^+$ ion on the time required for the reaction to proceed half-way to completion. It can be shown that for zero, first, and second order reactions, this time (denoted $t_{½}$), is expressed as follows:

$$t_{1/2} = \frac{C_0}{2k} \; for \; n = 0,$$

$$t_{1/2} = \frac{\ln 2}{k} \; for \; n = 1,$$

$$t_{1/2} = \frac{1}{C_0 k} \; for \; n = 2.$$

In general, the half-life is proportional to $(C_0)^{1-n}$. We will use these relations to determine the order of the reaction in Part I. Because the rate constant can be readily calculated from $t_{½}$ as shown above (they are inversely proportional), a determination of $t_{½}$ will yield the rate constant.

Question A: What would you expect for the ratio of the half-lives, $t_{½}$ (0.05 M) / $t_{½}$ (0.01 M), if the reaction were (a) zero order, (b) first order, or (c) second order?

The last part of the assignment is the determination of the activation energy of the reaction. The rate constant for a reaction is related to the energy of activation, E_a, by the Arrhenius equation:

$$k = Ae^{-E_a/RT}$$

where A is a constant characteristic of the reaction, R is the gas constant, and T is the absolute temperature in Kelvin. By taking the logarithm of both sides, we obtain:

$$\ln k = -\frac{E_a}{R}\left(\frac{1}{T}\right) + \ln A$$

Thus, if you determine the rate constant for the aquation of the chloro complex at several different temperatures, you can make a plot of ($\ln k$) vs. ($1/T$). A straight line drawn through the points should have a slope of $-E_a/R$, and so a determination of the slope permits a calculation of E_a.

Food Spoilage Prevention

A common example of chemical kinetics is the browning of an apple slice. This color change is caused by a reaction between enzymes present in the apple and oxygen from the the air. Enzymes are proteins that help catalyze (speed up) chemical reactions in metabolic processes. To slow down this process, we can manipulate the enzyme kinetics by storing food in the refrigerator. This is because the rate of chemical reactions decreases as temperature decreases. The Arrhenius equation is a mathematical equation that offers a calculated and proven explanation of the relationship between temperature and the rate of a chemical reaction. Thus, the lower temperature setting in a refrigerator can effectively slow down the rate of oxidation reaction and prevent the apple from browning rapidly.

Learning Goals

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

Laboratory	• Measuring rates and temperatures • Dilutions • Synthesizing and isolating different isomers
Conceptual	• Basic rate law principles • Activation Energy • Arrhenius Equation • Half-life
Data Analysis	• Determining the order of reactions calculations • Determining activation energy • Arrhenius plots

Procedure

You will **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.

Safety First

Wear your goggles and gloves throughout this experiment.

Some of the reagents used in this experiment can cause burns or other skin damage.

Stock Chemicals Used

Chemical	Maximum Amount Used
3 M Sulfuric Acid	10 mL
trans-$[Co(en)_2Cl_2]Cl$	0.5 grams

Part I. Determining Order of the Reaction (by using $t_{½}$)

1. Prepare the dilute sulfuric acid solvent:

   You should have ~60 mL of 0.3 M sulfuric acid from last week's experiment. If you no longer have the solution, prepare 60 mL of 0.3 M sulfuric acid solution by mixing 6 mL of a 3 M H_2SO_4 solution with 54 mL of deionized water.

2. Prepare a stock acid solution of the green chloro complex, about 0.050 M in *trans*-$[Co(en)_2Cl_2]Cl$.

   Dissolve about 170 mg of the compound measured precisely in 12 mL 0.3 M sulfuric acid solution in a 100 mL beaker. Store this solution in an ice-water bath.

3. Using a disposable pipet, transfer exactly 1.5 mL of the stock 0.050 M green chloro solution to one test tube and 3.0 mL to each of two other test tubes. Store the 3.0 mL solutions in an ice-water bath.

Lab Skill Tips

In order to get correct color matches, it is important to use the same size test tubes for all tests in Parts I and II.

4. Prepare a similar but more dilute green chloro solution, about 0.010 M in the green chloro complex.

   Dissolve about 33 mg of *trans*-$[Co(en)_2Cl_2]Cl$ in 12 mL of the 0.3 M sulfuric acid solution. Store this solution in an ice-bath.

5. Using a disposable pipet, transfer one 1.5 mL and two 3.0 mL portions of the 0.01 M solution to separate test tubes. Store the two 3.0 mL solutions in an ice-bath.

6. Heat the two test tubes containing 1.5 mL of solution in a beaker of boiling water using a hot plate for 5 to 10 minutes. This treatment will

affect complete conversion to the red aquo complex. Cool these two test tubes to room temperature, and to each solution add 1.5 mL from the two stock solutions you prepared containing the corresponding concentration of the original complex. Store both test tubes in ice-water bath.

7. You now should have six test tubes, each containing 3 mL of solution (see Table I). There are two sets of three tubes; the concentration in one set of solutions is about five times that of the other set. Two members of each set contain a green solution of essentially pure *trans*-$[Co(en)_2Cl_2]Cl$. The third tube of each set contains a grayish-pink solution, half of which has been converted to the aquo complex. Keep these two grayish-pink standards in an ice bath so that further conversion from chloro to aquo is minimized.

Table I.

Test Tube #	Contents	Brief Procedure
1	1.5 mL 0.05 M *trans*-$[Co(en)_2Cl_2]Cl$	boil—turns red; + 1.5 mL 0.05 M *trans*-$[Co(en)_2Cl_2]Cl$ *3 mL grayish $t_{1/2}$ comparison standard for 0.05 M runs*
2	3.0 mL 0.05 M *trans*-$[Co(en)_2Cl_2]Cl$	0.05 M run #1
3	3.0 mL 0.05 M *trans*-$[Co(en)_2Cl_2]Cl$	0.05 M run #2
4	1.5 mL 0.01 M *trans*-$[Co(en)_2Cl_2]Cl$	boil—turns red; + 1.5 mL 0.01 M *trans*-$[Co(en)_2Cl_2]Cl$ *3 mL grayish $t_{1/2}$ comparison standard for 0.01 M runs*
5	3.0 mL 0.01 M *trans*-$[Co(en)_2Cl_2]Cl$	0.01 M run #1
6	3.0 mL 0.01 M *trans*-$[Co(en)_2Cl_2]Cl$	0.01 M run #2

8. Adjust the temperature of a 250 mL beaker containing 150 mL water to within at least 2 °C of 55 °C. Take two test tubes of the essentially pure green chloro complex—one from the 0.05 M set, and one from the 0.01 M set (i.e. Test Tube #2 and 5). Place the two tubes into the beaker of hot water, noting the time to the nearest second.

   At the beginning, agitate the two test tubes in the hot water bath so that temperature equilibrium is rapidly attained (be careful not to break the thermometer). Maintain the bath temperature constant to ±0.5° by adjustment of the hot plate or, if necessary, by addition of small amounts of cold water. Record the exact temperature of the bath. Record the times at which the color of each solution matches the grayish-pink color of the corresponding half-reacted solution (i.e. Tubes #1 and 4). For best

Safety First

Be careful when using the hot plate and boiling water. Use the test tube tongs to hold the test tubes.

comparison, briefly lift the test tube out of the hot water bath and use a white paper background.

9. Repeat the experiment with the next two test tubes for trial #2. Average the results for each concentration from the two runs and determine the associated errors.

10. Save the test tube containing the 0.05 M reference half-reacted complex (i.e. test tube #1) for Part II.

Part II. Determination of the Activation Energy

1. You have already determined $t_{½}$ for a temperature near 55°C in Part I. However, the measurement was not held truly constant at 55°C for the entire duration of the reaction time since the test tube was originally at room temperature and thus required some time to equilibrate to 55°C in the water bath. For this section, in an effort to get a more accurate $t_{½}$ determination, you will add solid directly to pre-equilibrated dilute acid solution in test tubes already at the prescribed water bath temperatures.

2. Prepare six clean test tubes each filled with 3 mL portions of the 0.3 M sulfuric acid solution you made for Part I.

3. Weigh into a boat 40 mg of *trans*-$[Co(en)_2Cl_2]Cl$ for reference. Place the reference at your workstation alongside your bulk supply of green complex. As each trial is performed scoop out roughly 40 mg by gauging it visually against your reference.

Question B. Why is the precise mass not important?

Lab Skill Tips

Be organized to collect data at different temperatures.

Use test tube holders to hold the test tubes when working with boiling water.

4. Since you will be working in pairs for this experiment, one student should collect data at 80, 75, and 55 °C while the other student should collect at 80, 65, and 45 °C, each according to the following procedure. Both students should measure $t_{½}$ at 80 °C since the reaction is very fast at that temperature and the results may vary.

   Adjust the water bath to the desired temperature with the test tube already in the water bath. After the test tube containing the 0.3 M sulfuric acid has completely equilibrated (5–10 min.), quickly transfer a previously weighed portion of *trans*-$[Co(en)_2Cl_2]Cl$ into the test tube and swirl while holding it inside the water bath to insure complete dissolution of the solid. Compare the color with that of the half-reacted reference test tube referred to in Step 10 of Part I to determine the $t_{½}$ at that temperature. Time how long it takes mixture to reach the desired "gray" color.

5. Each pair of students should exchange data so that both of you have $t_{½}$ at five temperatures. For $t_{½}$ at 80 °C use the average value of data you and your partner collected.

Clean-up

- The cobalt **solutions** in the test tubes must be discarded in the waste container labeled **cobalt complex solution waste** found in the fume hood.
- Neutralize excess sulfuric acid with **0.5 gram of sodium bicarbonate** before sink disposal with copious amount of water
- Turn in any **solid** green cobalt complex to your TA.

Part I.

Question C: From your measured half-lives, you should be able to state the dependence of the half-life on the concentration of *trans*-$[Co(en)_2Cl_2]^+$.

Question D: Determine the order of your reaction and discuss any potential sources of error.

Part II.

Question E: For each of the five temperatures, calculate the rate constant and then plot ln *k* as a function of 1/T. What is the energy of activation for the reaction?

Question F: What is the value of the rate constant at 25 °C?

Question G: As stated in the introduction, a plot of ln k as a function of 1/T yields a straight line with slope $-E_a/R$. Show that a plot of ln $t_{½}$ versus 1/T would have a slope of $+E_a/R$ given that for reactions of order zero, one, or two; and $t_{½}$ is inversely proportional to *k*. Does the proportionality constant affect the slope?

Question H: For many reactions near room temperature, the rate approximately doubles for a 10° C rise in temperature. What is the corresponding activation energy?

Supplementary Activities (9 points).

Determining Order of the Reaction (by tracking the reaction over time)

1. On-line you will find a set of data collected on a solution of the green chloro complex that was monitored over time during the course of its conversion to the aquo complex. Use these data of Time vs. Concentration of green complex and the integral forms of the rate laws to determine the order of the reaction. Hint: You will need to plot $[A]_t$ vs. time, $\ln[A]_t$ vs. time, and $[A]_t^{-1}$ vs. time. Plot all 3 graphs and determine which one yields a straight line. Use Excel.

2. On-line you will find a set of experimental concentration data for two different reactions, A & B. In each case, use these data of Time vs. Concentration for each reaction and the integral forms of the rate laws to determine the order of the reaction.

Turn in your 9 correctly labeled graphs to your TA.

Data Analysis

1. What values would you expect for the ratio of half-lives for a reaction with starting concentrations of 0.05 M and 0.01M, $t_{½}$ (0.05 M) / $t_{½}$ (0.01 M), if a reaction is known to be zero order?

2. What values would you expect for the ratio of half-lives for a reaction with starting concentrations of 0.05 M and 0.01 M, $t_{½}$ (0.05 M) / $t_{½}$ (0.01 M), if a reaction is known to be first order?

3. What values would you expect for the ratio of half-lives for a reaction with starting concentrations of 0.05 M and 0.01 M, $t_{½}$ (0.05 M) / $t_{½}$ (0.01 M), if a reaction is known to be second order?

4. What value do you calculate for the ratio $t_{½}$ (0.05 M) / $t_{½}$ (0.01 M) from your experimentally measured half-lives at 55 °C?

5. Given your experimentally measured ratio of half-lives for the two different concentrations, what is the concentration dependence that best describes your experimental ratio.

6. Based on the ratio of half-lives for the two different concentrations that you determined, and comparing the experimental ratio with the theoretical ratios for kinetic orders 0, 1, and 2, which order does this reaction exhibit?

   The following series of questions pertains to the analysis of the data in Part II of the Kinetics Experiment. The activation energy of the reaction will be determined based on your data. Your data and resulting calculations will be verified as you proceed through the exercise.

7. In part II of the experiment you and your partner measured the half-life of the reaction at 5 different temperatures. You should have constructed a table with column headings T(°C), T(K), 1/T, $t_{½}$, k = ln(2)/$t_{½}$, ln(k) and rows for each of the five temperatures for which your team determined the half-lives. If you have not completed such a table, either extend your existing table to include all these columns, or create the entire table now.

8. Record the 5 precise temperatures in degrees Celsius at which you measured the half-life of the reaction (e.g. 81.5). Record them in increasing order (lowest to highest).

9. Convert each of the temperatures at which you measured the half-life of the reactions to Kelvin and find the reciprocal.

10. Record the 5 measured half-lives in **units of seconds** in corresponding order with the Celsius temperatures.

11. Calculate the rate constant of each of the half-lives, $t_{½}$. Record the rate constants in the corresponding order with the of the half-lives. Report your rate constant values using at least 5 significant figures.

12. Calculate the natural logarithm of each of the rate constants, k. Record the natural logarithm values in the corresponding order with the of the rate constants. Report your natural logarithm values using at least 5 significant figures.

13. Plot the values of ln(k) vs. 1/T on a spreadsheet or a programmable calculator. Carefully, determine the slope of the best straight line fit through the data using a regression (least squares fitting) formula provided by your calculator or spreadsheet. The slope value should be accurately determined to enable you to calculate a reasonable value for the activation energy of the reaction. Remember that the best fit line of the points on this graph should not be forced to go through the origin, (0,0), since the y-intercept in not necessarily zero. Record the value of the slope of ln(k) vs.1/T.

14. The activation energy of the reaction may be calculated using the previously determined slope. Calculate for the activation energy in kJ/mol using the value you obtained for the slope of ln(k) vs. 1/T.

15. Use one of your experimentally determined values of k, the activation energy you determined, and the Arrhenius equation to calculate the value of the rate constant at 25 °C. Alternatively, you can simply extrapolate the straight line plot of ln(k) vs. 1/T in your notebook to 1/298 K, read off the value of ln(k), and find the inverse of ln k.

    k(25 °C) = _____

16. By showing the algebra for combining the Arrhenius equation with the expression for the half-life of a reaction of order zero, one or two, you should convince yourself that a plot of ln($t_{½}$) vs.1/T will have a slope of $+E_a$ / R as long as the half-life is inversely related to the rate constant. Use this information to answer the following question. Does the proportionality constant between half-life and inverse rate constant affect the slope of a plot of ln($t_{½}$) vs. 1/T?

17. For many reactions near room temperature, the rate and the rate constant approximately doubles for a 10 °C rise in temperature. What is the value of activation energy in kJ/mol for such a reaction?

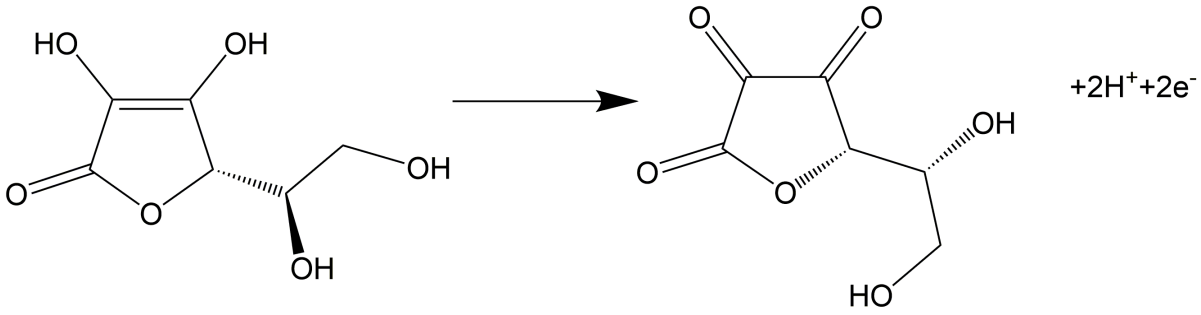
18. Listed below are some data that was collected on a solution of the green chloro complex as a function of time during its conversion to the aquo complex. Copy these data into your notebook or cut and paste these values into a spreadsheet program. Use this data and the integrated forms of the rate laws to determine the order of the reaction. You must plot all three graphs to determine which yields a straight line. The plot that gives a clear straight line identifies the order of the reaction. Report the order of the reaction and turn in your graphs to your TA at the beginning of the next laboratory period. Each graph is worth 1 point.

Determination of Vitamin C Content Via a Redox Titration

Introduction

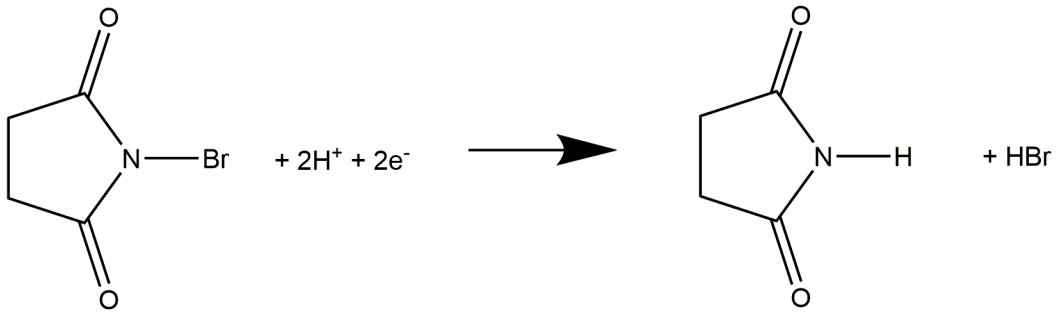
Vitamin C has received much publicity in the late 20th century as a promising antioxidant and a potential cure for the common cold according to the controversial hypothesis by the Nobel Laureate Linus Pauling. A more widely accepted role of Vitamin C is as a cure for one of the most suffered nutritional deficiencies, scurvy, which was especially threatening for sailors on long voyages in the 18th century. Vitamin C is chemically known as L-ascorbic acid, and human beings do not possess the biochemical metabolic pathways to produce it in the body. We must therefore consume Vitamin C (current daily recommendation is 60 mg) from various foods. It is found in many fresh fruits and vegetables such as sweet peppers, spinach, and citrus juices.

In this experiment, you will determine the Vitamin C content in various Vitamin C tablets readily available to the consumer at neighborhood drug stores. This determination will utilize the fact that L-ascorbic acid is readily oxidized to L-dehydroascorbic acid according to the following oxidation half-reaction (which has an oxidation cell potential of 0.127 V at pH = 5):



L-ascorbic acid L-dehydroascorbic acid

You will use another organic molecule, N-bromosuccinimide (NBS), as the oxidizing agent. The pertinent reduction half reaction is:



N-bromosuccinide (NBS) succinimide

The "indicator" in the "redox" titration you will be doing is actually generated in place by a second redox reaction after all of the ascorbic acid in the sample of interest has been consumed. The titration flask initially contains some added KI and starch solution. The first small excess of NBS oxidizes some iodide ions to molecular iodine, which then reacts with more I^- to produce the triiodide ion, I_3^-. The polymeric amylose (starch) molecules wrap themselves around the triiodide ions and form a blue colored aggregate to signal the endpoint.

Ascorbic acid solutions are susceptible to air oxidation, which can be inhibited in various ways, including acidification. The air oxidation of ascorbic acid is catalyzed by metal ions increasing the rate of degradation of ascorbic acid significantly. Oxalic acid forms complexes with most metal ions and prevents their functioning as an oxidation catalyst. Oxalic acid is commonly employed to increase the longevity of ascorbic acid. The last segment of this experiment will test these factors involved in the degradation of ascorbic acid.

Please read the appropriate chapter in your textbook as pre-laboratory preparation for this experiment.

The Many Roles of Vitamin C

Vitamin C, scientifically known as L-ascorbic acid, is an essential nutrient that plays a crucial role in your body. It helps form blood vessels, bone marrow, muscle, and collagen in skin and bones. It is also vital to the body's healing process and acts as an "antioxidant."

Free radicals are molecules with an unpaired electron and are highly reactive. They can form when cells are exposed to various factors, such as sun exposure, smoking, or the breakdown of food. When produced in excess, free radicals can damage cells and contribute to the development of diseases like cancer and heart disease and the premature aging of the skin. Antioxidants, on the other hand, are molecules that safeguard your cells against the damaging effects of free radicals. They donate electrons, neutralizing free radicals and reducing their ability to damage cells. This is why facial care products with vitamin C serums are highly sought after as they help reduce the appearance of wrinkles and age spots.

Remember to eat plenty of vitamin C-rich foods to fortify your body's defense against free radicals!

Learning Goals

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

Laboratory	• Iodine titrations • Making a standard and analytical solution
Conceptual	• Oxidation/reduction • Redox reactions with organic compounds • Balancing half-reactions • Alcohol oxidation to a ketone • Chemical degradation • Determining content of desired produt in a mixture
Data Analysis	• Standardizing solutions • Using titrations to find unknown concentrations • Determining unknown mass and composition • Determining total degradation of a sample

Procedure

You will **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 Acetic Acid	6 mL
1% Starch	8 mL
Oxalic Acid, (Solid)	4 grams
Ascorbic Acid, (Solid)	0.2 grams
4% Potassium Iodide solution, 1L	40 mL
N-Bromosuccinimide (NBS), (solid)	0.1 grams
Commercial Vitamin C tablets	0.1 grams
0.05 M Ferric Nitrate	2 mL

Safety First

Place all waste solutions/ precipitates into the proper waste container.

Wear your goggles!

Part I. Preparation of solutions

Your TA will place you in a group of four to prepare the necessary reagents for this experiment.

Titrant Solution. Prepare about 250 mL of an NBS titrant solution by dissolving approximately 100 mg of NBS in an Erlenmeyer flask.

Primary Standard. Accurately weigh ca. 100 mg of ascorbic acid and transfer it quantitatively to your 250 mL volumetric flask. Add ca. 2 g of oxalic acid to the flask, partially fill it with deionized water and swirl until the solids have dissolved.

Safety First

Do not invert or swirl the flask vigorously.

Continue gradually adding water and swirling the flask to keep the contents well mixed, and finally add water from a disposable pipet to bring the liquid level to the mark. Do a final mixing by inverting and swirling the flask at least 30 times. Keep the volumetric flask sealed as much as possible to minimize the opportunity for air oxidation to occur.

Analytical Sample. Follow a similar procedure as above but use an accurately weighed ca. 100 mg sample of a commercial Vitamin C tablet in place of the ascorbic acid.

You will first have to crush the Vitamin C tablet in a folded sheet of paper using a small iron ring as the hammer, making a fairly fine powder. Further

grind the crushed tablet using the pointed end of a centrifuge tube as a pestle and a small beaker as a mortar into a very fine powder. These tablets sometimes contain binders, which will not completely dissolve.

Solution with Metal Ions. Accurately weigh ca. 100 mg of ascorbic acid and transfer it quantitatively to a clean 250 mL volumetric flask. Partially fill the volumetric flask with deionized water and swirl until the solids have dissolved.

Continue gradually adding water and swirling the flask to keep the contents well mixed, and finally add water from a disposable pipet to bring the liquid level to the mark. Do a final mixing by inverting and swirling the flask at least 30 times. Divide the solution approximately in half into two 250 mL beakers.

To one beaker, add 1 g of oxalic acid and stir with a glass rod until the oxalic acid has dissolved. Label each of the beakers so that the one containing oxalic acid can be distinguished from the one without. Add 1 mL of 0.05 M ferric nitrate solution to each of the beakers and set them aside.

Part II. Titrations

Do these titrations one at a time—in other words, do not let ascorbic acid solutions sit around exposed to the atmosphere for very long.

Buret Usage Tips

- 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.

Assaying your NBS solution: With a volumetric pipet take a 10 mL aliquot of the "partnership's" standard ascorbic acid solution and add it to a 125 mL Erlenmeyer flask that already contains 20 mL of water, 1 mL of 6 M acetic acid, 5 mL of 4% KI solution and 1 mL of starch indicator solution.

As you approach the end point you should see the blue color form where the drop enters but go away again when the flask is swirled. Repeat this titration at least two more times.

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.

Clean-up

- Neutralize the titration flask with **0.5 gram of sodium bicarbonate** before sink disposal with copious amount of water.

Analyzing commercial Vitamin C: Perform the above titration procedure at least three times using the solution of the commercial Vitamin C tablet and thereby obtaining the data needed to compute the ascorbic acid content of this product.

Part III. Investigation of degradative factors on ascorbic acid

Effect of Metal Ions

Titrate a 10.00 mL aliquot of the ascorbic acid solution containing metal ions plus the oxalic acid. Follow the procedures given in Part II under Assaying your NBS solution. Repeat this titration to obtain more accurate results if there is time.

Titrate a 10.00 mL aliquot of the ascorbic acid solution containing metal ions without the oxalic acid. Follow the procedures given in Part II under Assaying your NBS solution. Repeat this titration to obtain more accurate results if there is time.

Clean-up:

- Combine the NBS solution and ascorbic acid solution, add **1.0 gram of sodium bicarbonate** before sink disposal with copious amount of water.

Data Analysis

Part I. Molarity of NBS Solution

1. Using the half reaction method, balance the redox reaction between one mole of NBS and ascorbic acid to give L-dehydroascorbic acid and succinimide. How many moles of electrons are transferred?

2. How many moles of NBS are consumed for every one mole of ascorbic acid reacted?

3. In the presence of excess iodide ions, the iodine formed by the reaction of iodide with NBS will react further to form triiodide ions. What does the triiodide combine with to form the blue color of the endpoint?

4. You were instructed to use approximately 100 mg of ascorbic acid to prepare your primary standard solution. What is the precise mass in milligrams of the ascorbic acid used to prepare your primary standard solution?

5. If one had weighed out precisely 100 mg of ascorbic acid for the primary standard solution and dissolved it in enough deionized water to make a 250 mL solution, the molarity of that solution would be 0.00227 M. What is your calculated molarity of the ascorbic acid primary standard solution based on the precise number of milligrams of ascorbic acid you weighed out?

6. In the standardization of the NBS solution using the ascorbic acid primary standard solution, your titration volumes of the NBS solution should be approximately 10-15 mL. For each trial, what is the precise volume in mL of NBS solution used in the titration of your ascorbic acid solution (e.g. 10.34 mL)?

7. Using the volumes of NBS solution you used to titrate the ascorbic acid primary standard solution and the molarity of the ascorbic acid primary standard solution, calculate the molarity of the NBS secondary standard solution for each trial. What is your calculated molarity of the NBS solution for each trial?

8. The molarity of the NBS solution is taken as the average of the three trials. What is the average?

9. What is the standard deviation of the molarities of the NBS solution?

Part II. Determination of Vitamin C Content

10. You were instructed to use approximately 100 mg of the Vitamin C to prepare your analytical sample solution. What is the value for the precise mass in mg of the Vitamin C tablet that was dissolved in your analytical sample solution?

11. In the analysis of the ascorbic acid content in a Vitamin C tablet, your titration volumes of the NBS solution should be approximately 5-10 mL. For each trial, what is the precise volume in mL of NBS used in the titration of the 10.00 mL Vitamin C sample (e.g. 8.93 mL)?

12. Using the volumes of NBS solution and the average molarity of the NBS standard solution, calculate the milligrams of ascorbic acid in the 10.00 mL Vitamin C sample for each trial.

What is your calculated milligrams of ascorbic acid in the 10.00 mL Vitamin C sample for each trial?

13. Using the milligrams of ascorbic acid in the 10.00 mL Vitamin C sample, the ratio of total sample volume to aliquot volume, and the total milligrams of the Vitamin C tablet that you dissolved, calculate the mass percent of ascorbic acid in the Vitamin C tablet for each trial. What is your calculated mass of ascorbic acid in the Vitamin C tablet for each trial?

14. Calculate the average mass percent of ascorbic acid in the Vitamin C tablet.

15. Calculate the standard deviation of the mass of ascorbic acid in the Vitamin C tablet.

Part III. Investigation of Degradative factors on Ascorbic Acid

In the final part of the experiment, you were asked to examine the protective ability of oxalic acid in preventing the oxidative degradation of Vitamin C by catalytic ferric ions.

At the beginning of the experiment you or one of your partners prepared two solutions containing ascorbic acid and ferric ions. In one of these solutions oxalic acid was added and the two solutions were set aside in open containers.

16. When you titrated a sample from each of these solutions, one containing oxalic acid and one without oxalic acid, which solution required more NBS to reach the endpoint in the titration?

17. For each trial, what is the precise volume in mL of NBS solution used in the titration of the ascorbic acid/ferric ion solution containing oxalic acid?

18. Using the volume of NBS required to neutralize the solution that DID contain oxalic acid and the molarity of your NBS solution, calculate the mass in mg of ascorbic acid present in the 10.00 mL sample solution that DID contain oxalic acid.

19. For each trial, what is the precise volume in mL of NBS solution used in the titration of the ascorbic acid/ferric ion solution that DID NOT contain any oxalic acid?

20. Using the volume of NBS required to neutralize the solution that DID NOT contain oxalic acid and the molarity of your NBS solution, calculate the mass in mg of ascorbic acid present in the 10.00 mL sample solution that DID NOT contain oxalic acid.

21. What is the mg of ascorbic acid that was protected by the presence of the oxalic acid?

22. Is oxalic acid effective in inhibiting the catalytic effect of metal ions? What redox reaction schemes may be responsible for the direct degradation of ascorbic acid?

23. What redox reaction schemes may be responsible for the catalytic degradation of ascorbic acid?

24. What transition metal structures involving oxalate ion (from the oxalic acid) and iron would you expect to be responsible for the inhibition of catalytic degradation of ascorbic acid?

25. Vitamin C can be added to applesauce to inhibit it from becoming brown in color when exposed to air. Discuss how this protection occurs based on the reactions of Vitamin C and its possible role as a sacrificial antioxidant.

Conclusion.

Briefly discuss interpretations of your observations and results. Include in your discussion, any conclusions drawn from the results and any sources of error in the experiment.

Chem 2 Series Laboratory

Procedures and Safety Handbook

Revision Date: June 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.

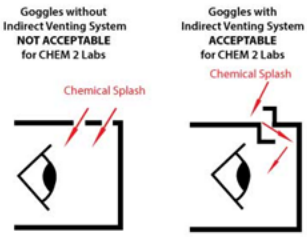
  A good rule of thumb to keep in mind is: **No skin exposure from the neck down to the feet in the laboratory.**

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.

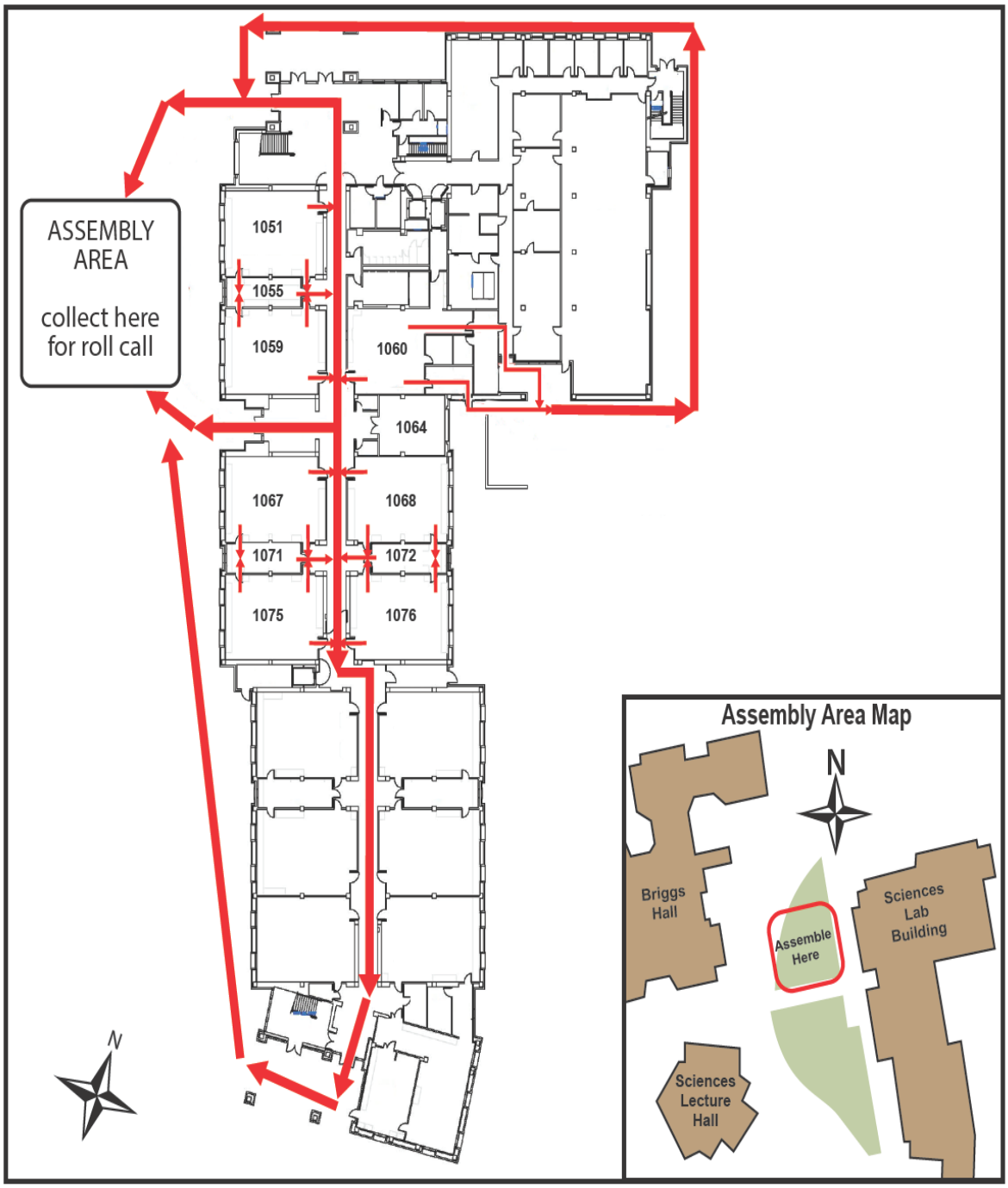


Figure 1. Evacuation routes for the 1st floor SLB rooms.

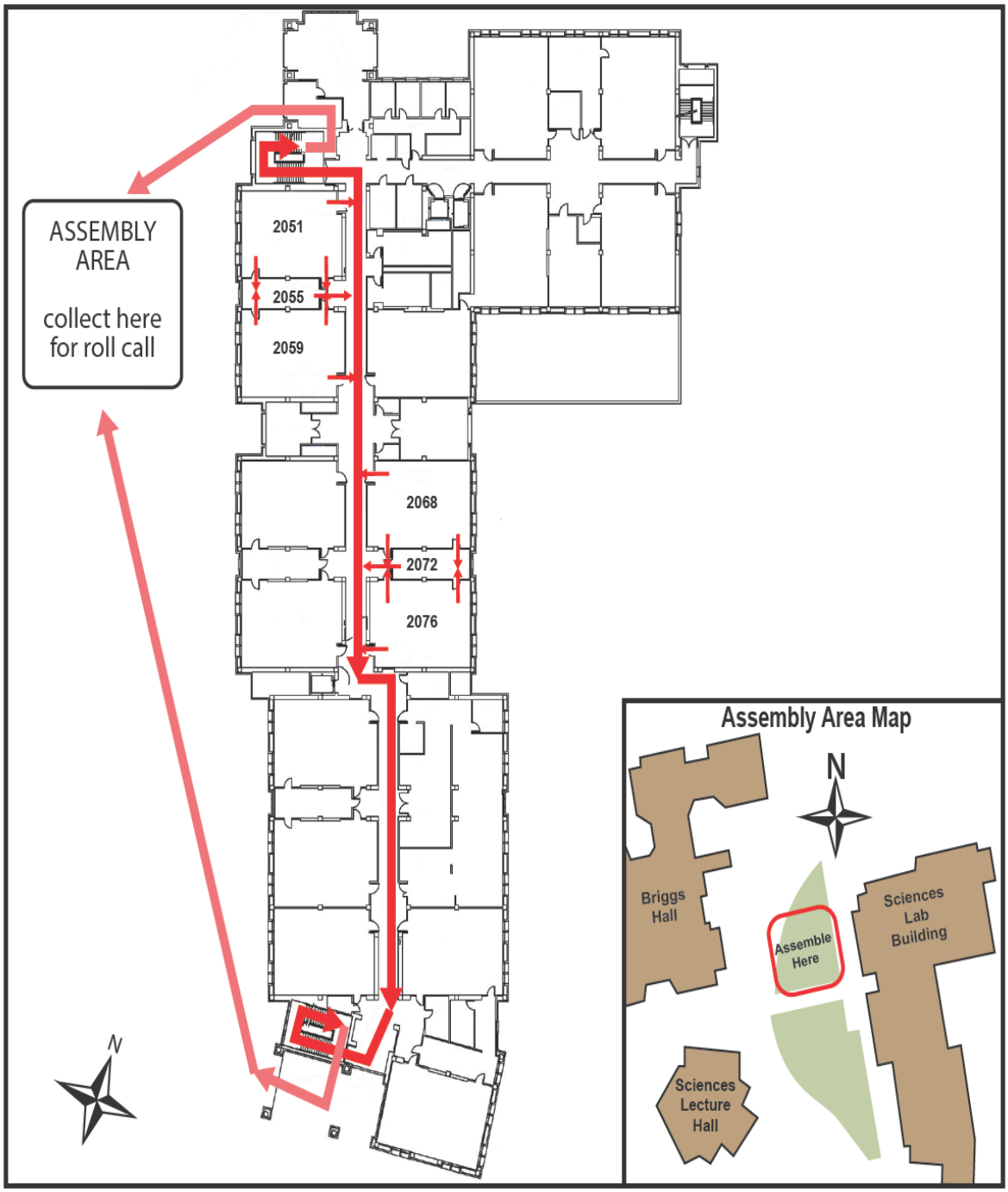


Figure 2. Evacuation routes for the 2nd floor SLB rooms.

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 Sciences Lab Building.
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 SLB 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.

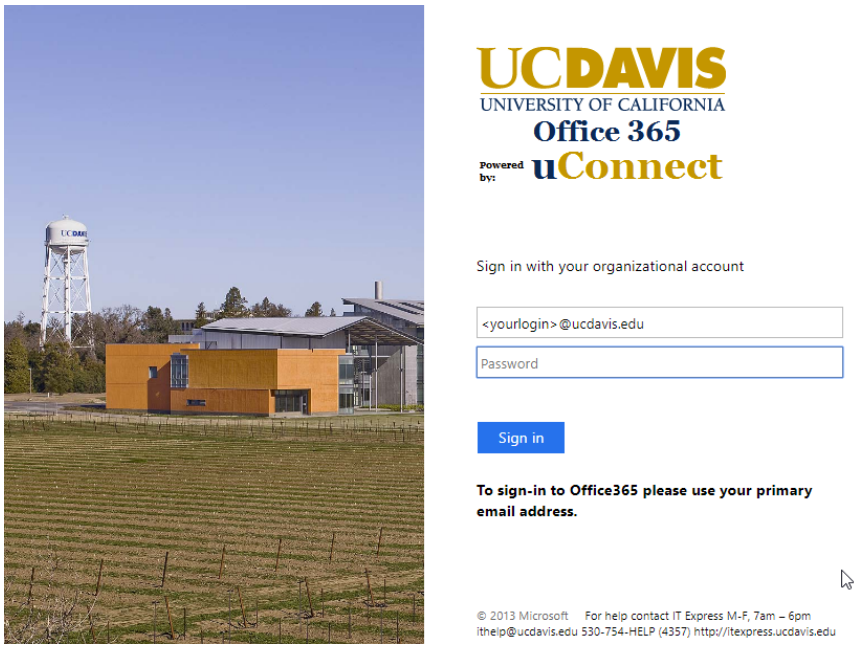


Figure 1. Use your UC Davis login information to access Microsoft Office 365.

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.

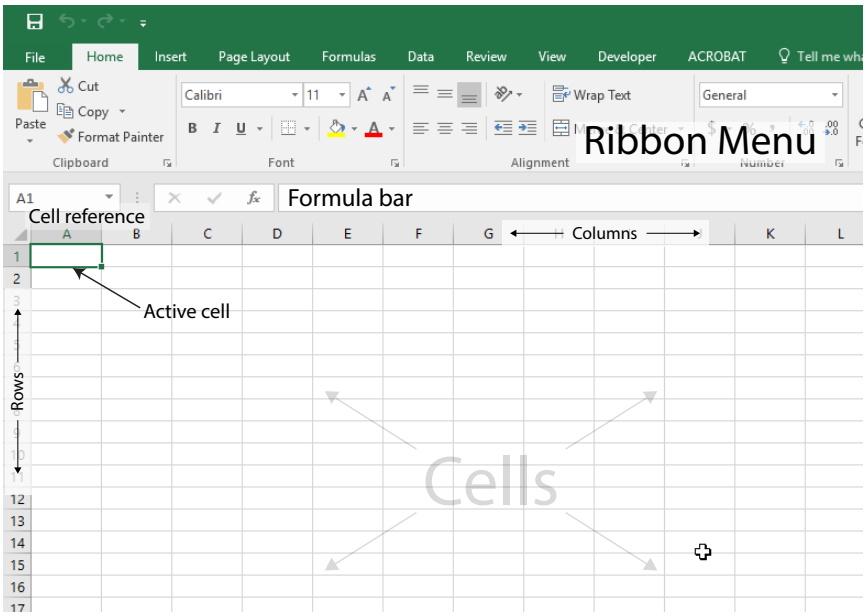


Figure 3. A blank spreadsheet in Excel 2016.

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.

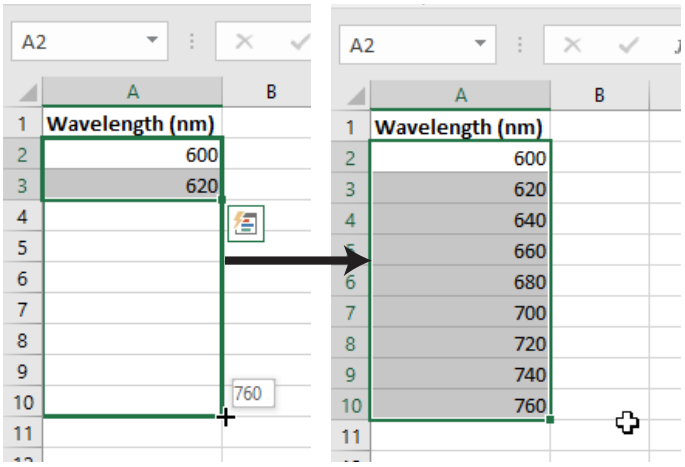


Figure 5. Using the Fill Handle to expand a series of increasing wavelengths.

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.

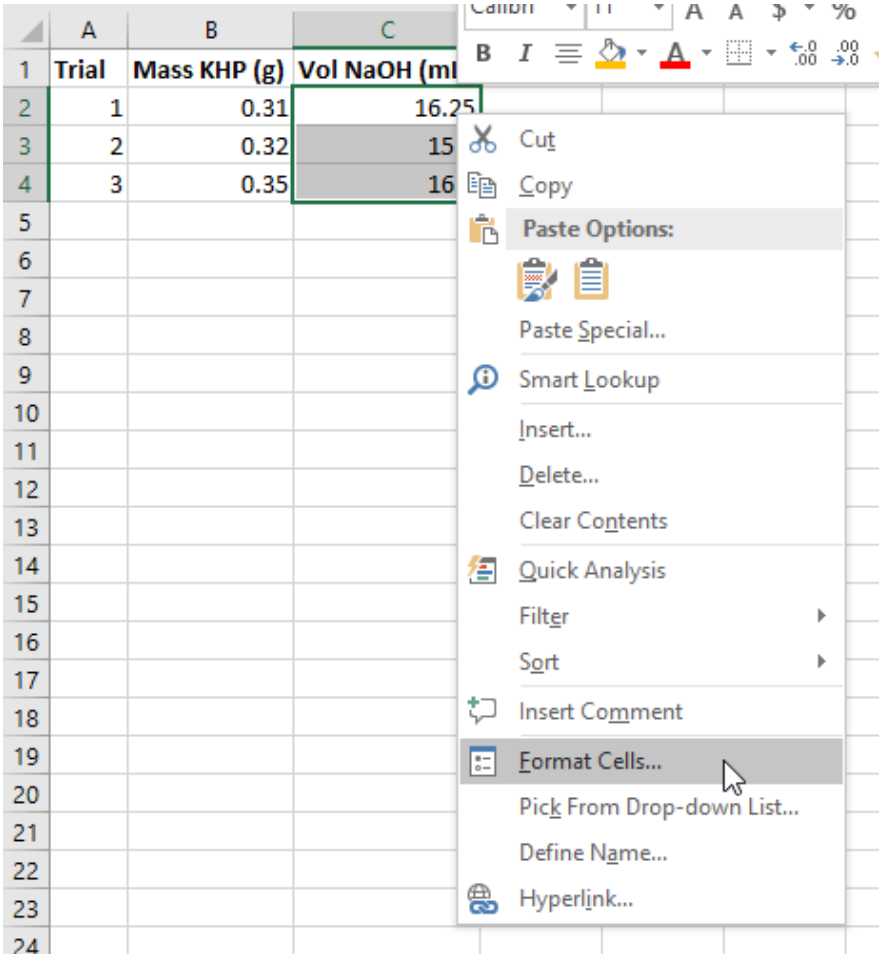


Figure 6. Select Format Cells... from the context menu.

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.

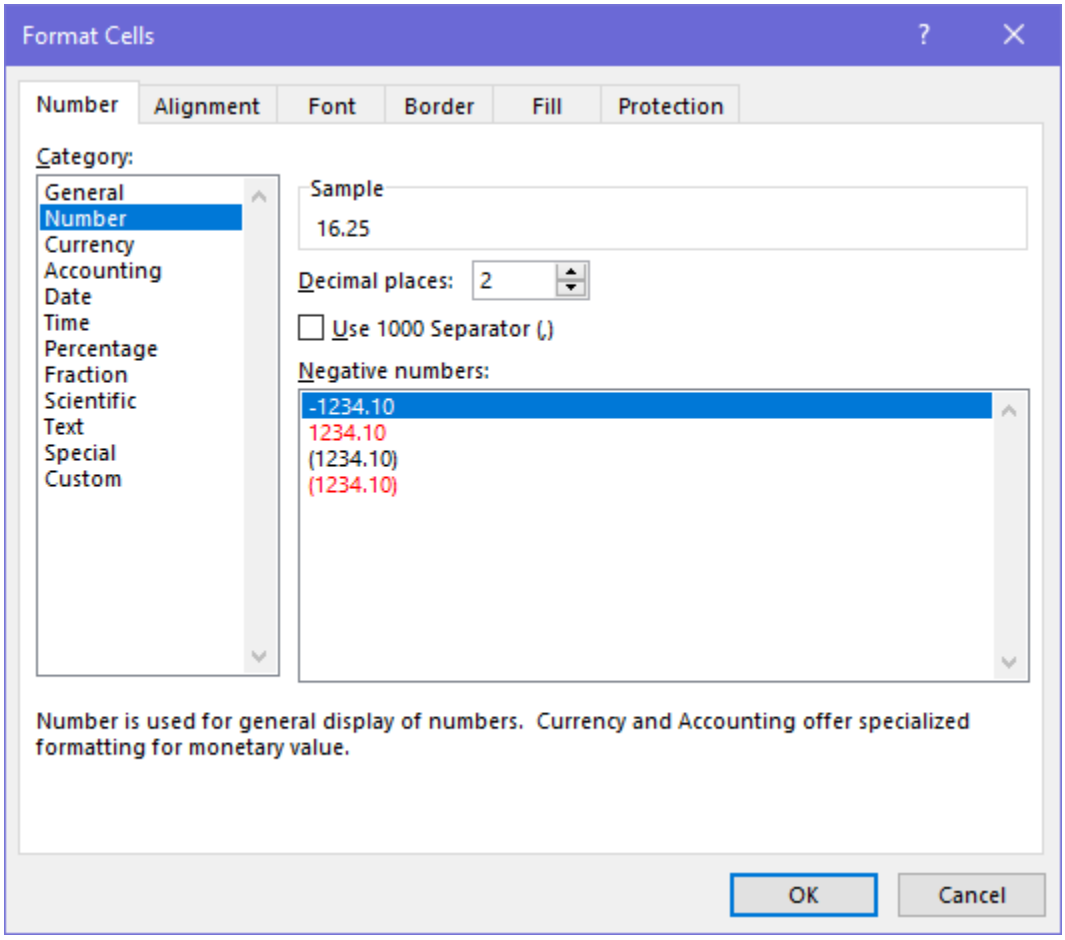


Figure 7. The Format Cells window showing the Number formatting.

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.

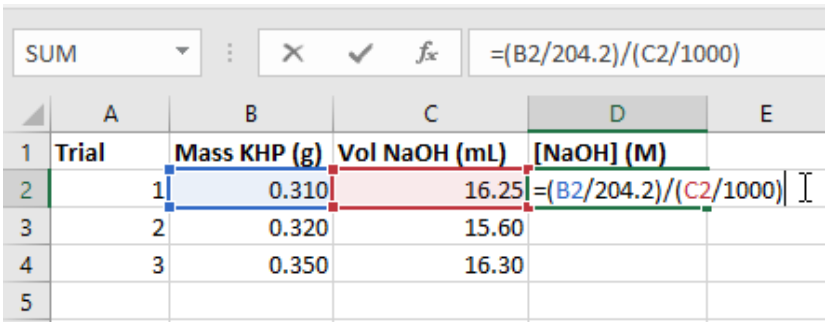


Figure 8. The equation typed into cell D2 as an Excel formula.

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.

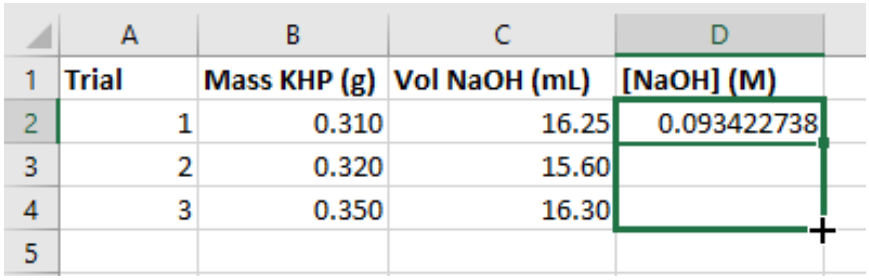


Figure 9. Click and drag the handle down to the last row of data.

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.

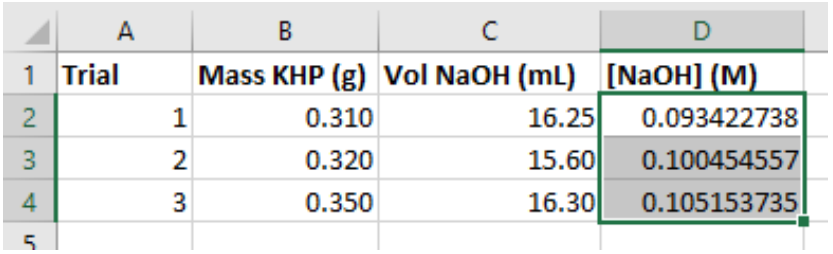


Figure 10. The formula bar showing the updated cell references.

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.

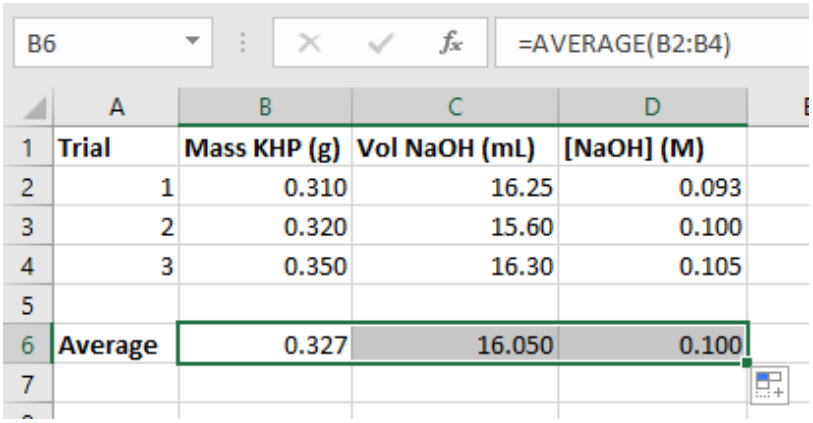


Figure 11. The formula bar shows the formula used to calculate the value in the cell.

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.

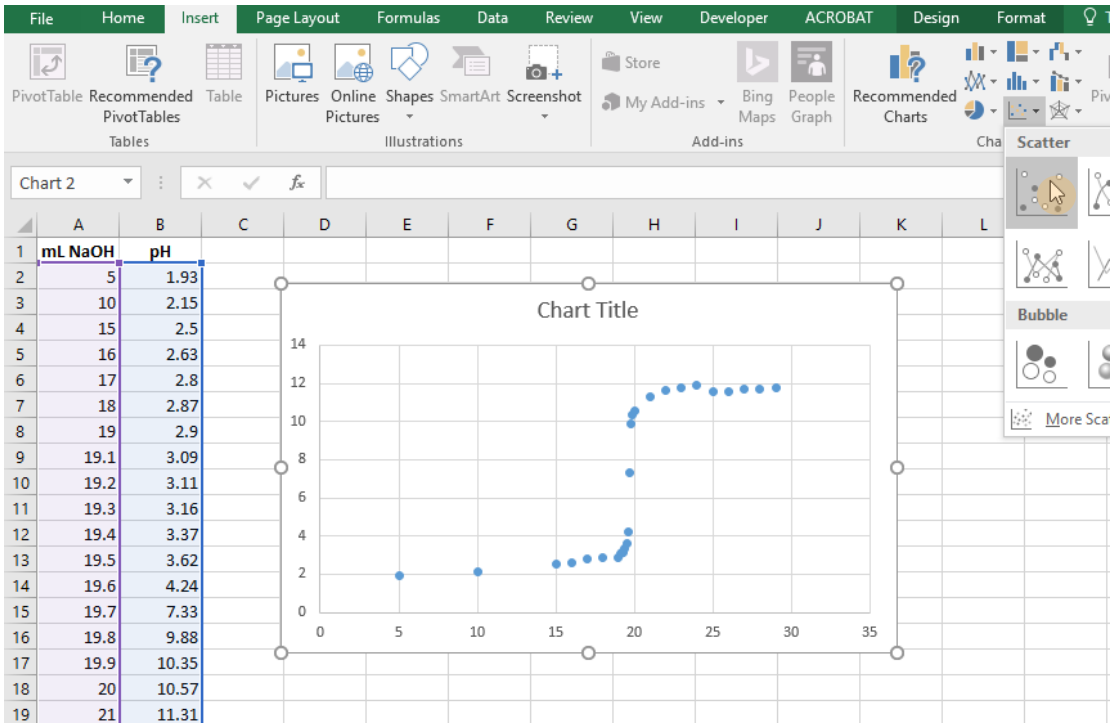


Figure 12. After selecting the data range, go to Insert > Scatter to plot the points on a graph.

   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.

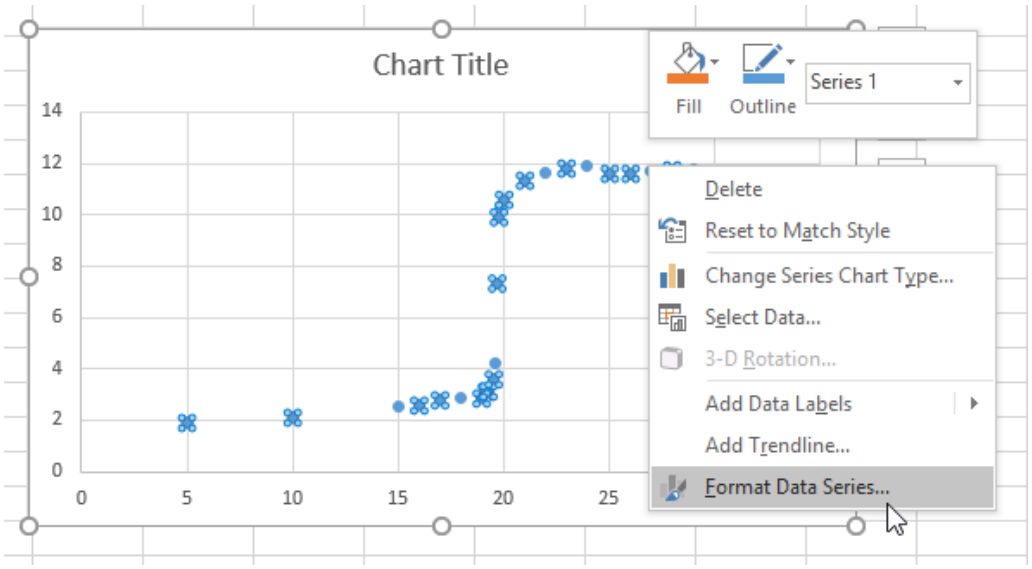


Figure 13. Select Format Data Series from the context menu to access more options.

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**.

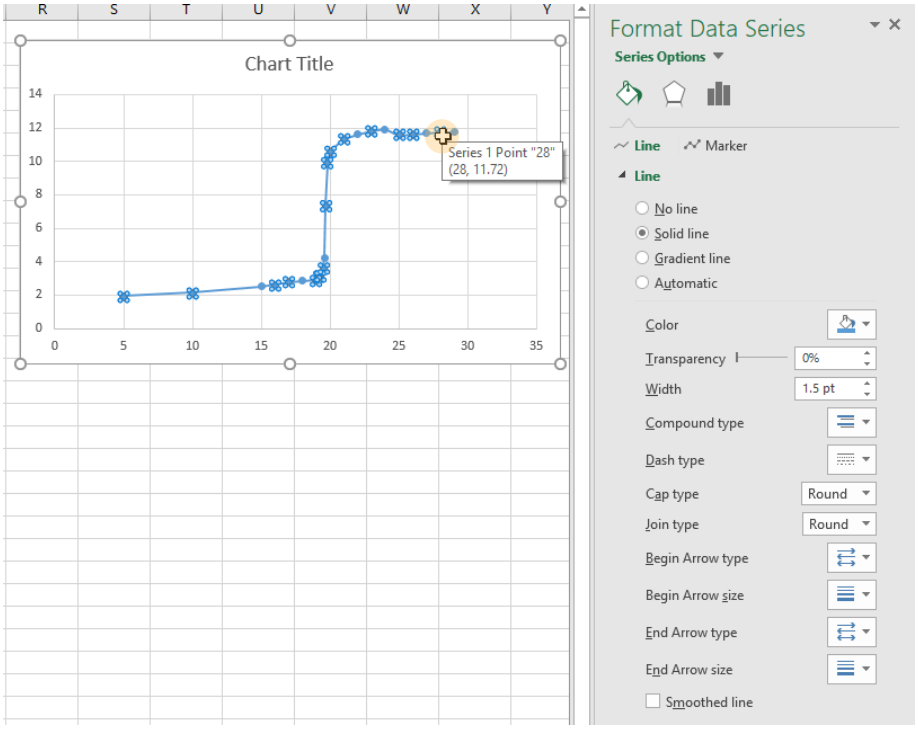


Figure 14. The titration curve with connecting lines added.

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.

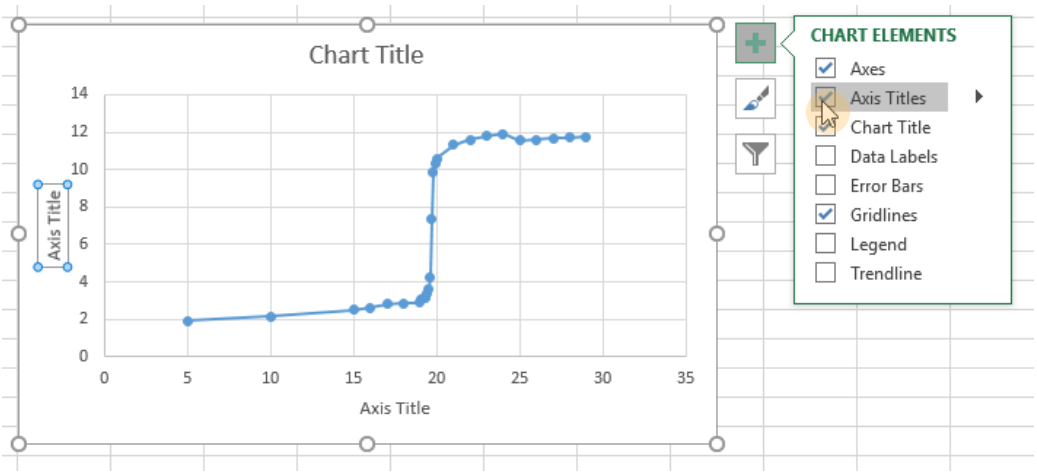


Figure 15. The Chart Elements menu.

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.

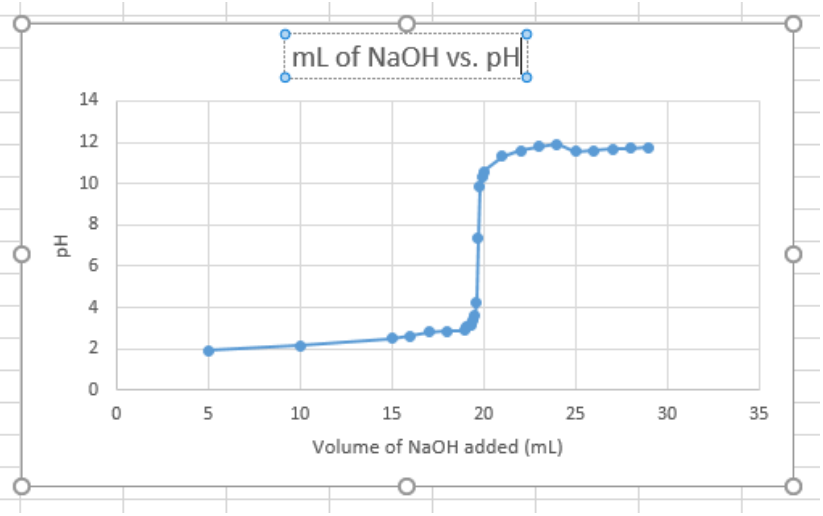


Figure 16. The titration curve with a title and axis labels added.

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.

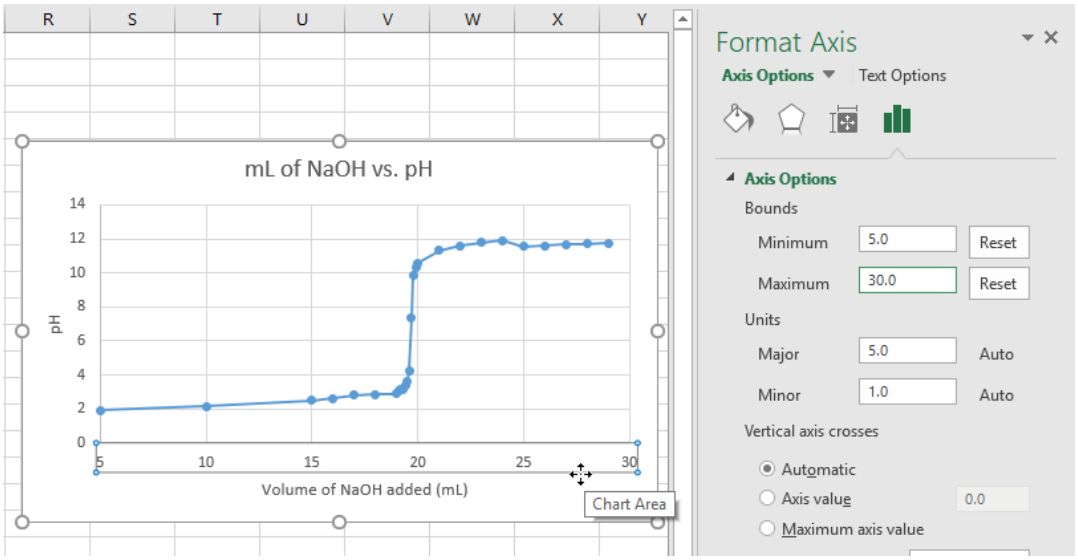


Figure 17. Changing the minimum and maximum bounds of the x-axis.

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.

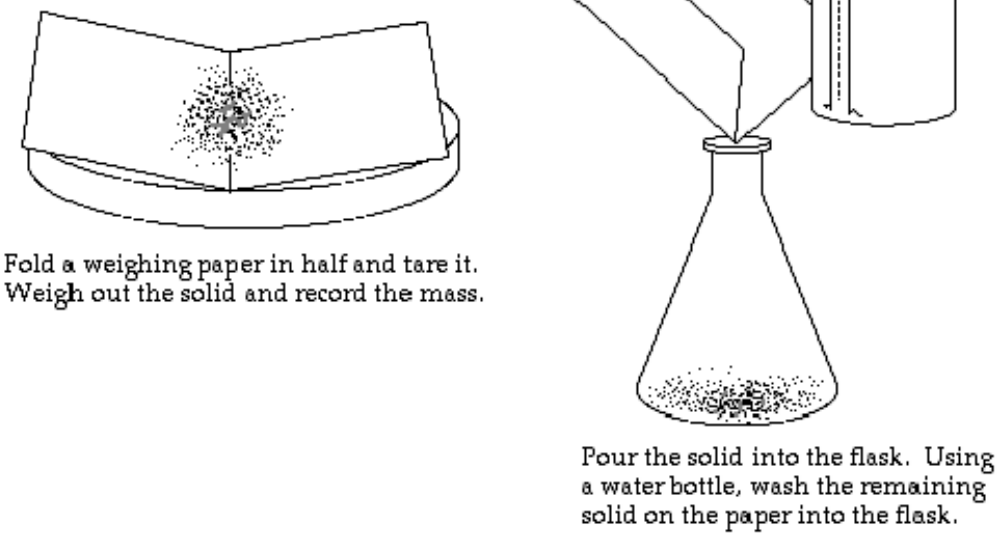


Figure 1. Quantitative Transfer of Solids

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

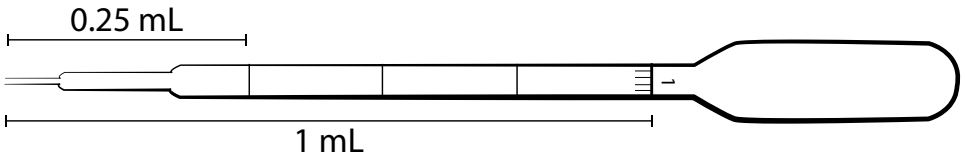
- Improper use of disposable pipets may cause serious injuries!
- Never point the pipet at yourself or others!
- Do not squeeze air into solutions with the dispo pipet. This may result in chemical splashes.
- Always put full dispo pipet in a test tube when carrying it to another part of the lab.

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.

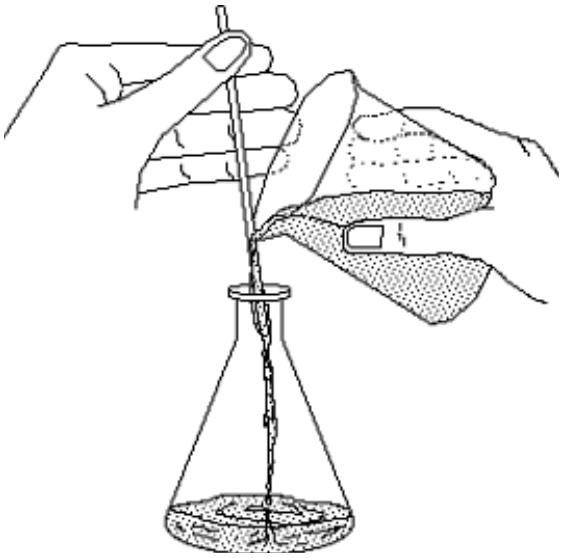


Figure 2. Liquid Transfer with Glass Stir Rod

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.

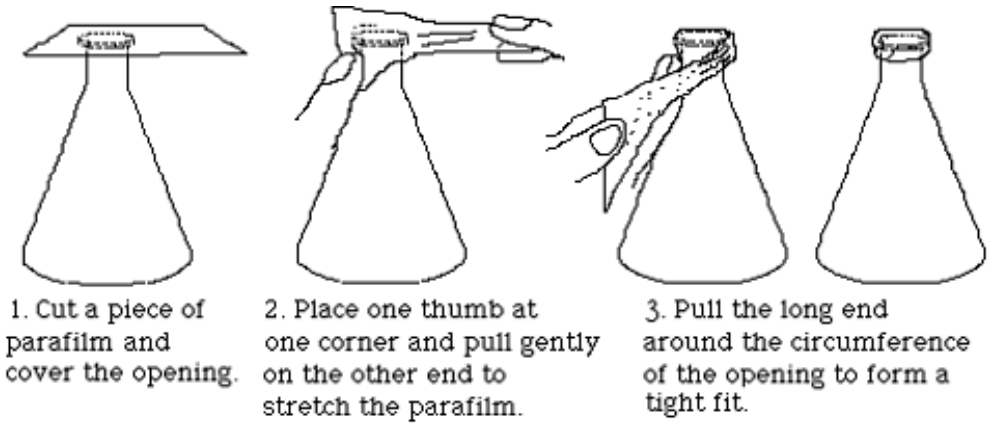


Figure 3. Capping A Flask

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.

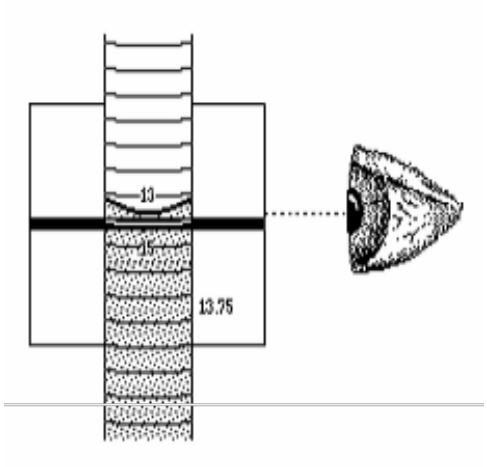


Figure 4. Reading the Meniscus

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.

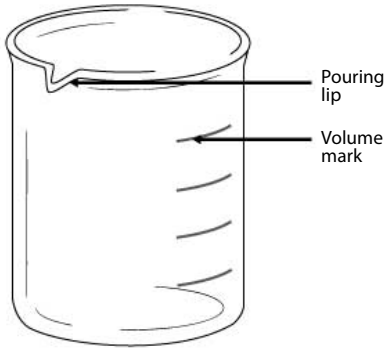
a. Always hold beakers from the bottom or the side. Never hold a beaker by the rim.

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!**

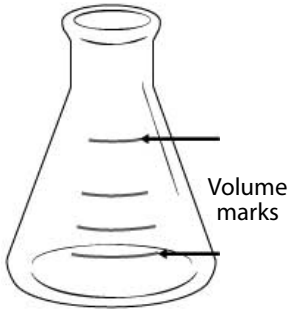


3. Erlenmeyer Flasks

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!**

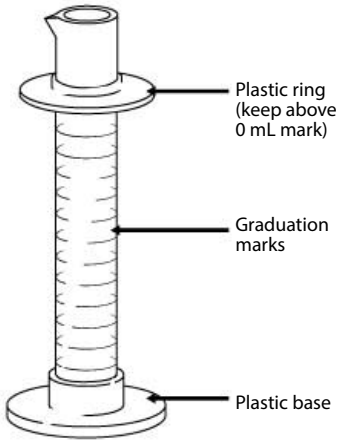


4. Graduated Cylinder

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.



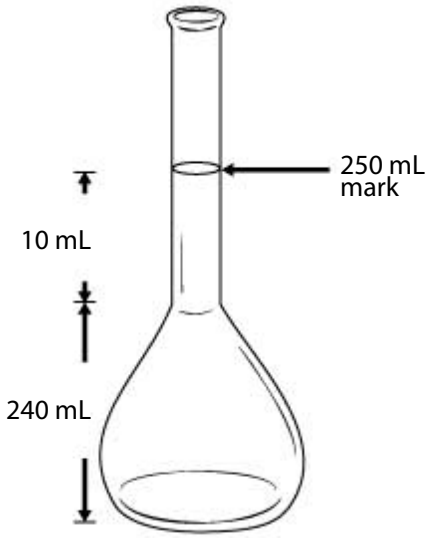
5. Volumetric Flasks

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.

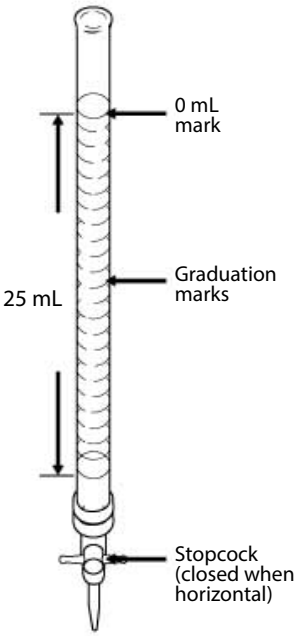
b. To fill a volumetric flask to the mark, quickly fill the flask to where the round base meets the neck. Cap the bottle and swirl or invert if needed. Then use a **250 mL water squeeze bottle** to fill to the volume mark. Notice that the volume between the neck and the 250 mL volume mark is only 10 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

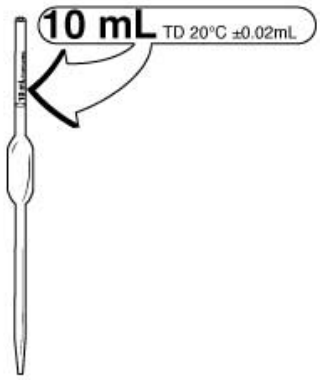
Similar to the buret, the volumetric pipet is designed to deliver a precise amount of solution.

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

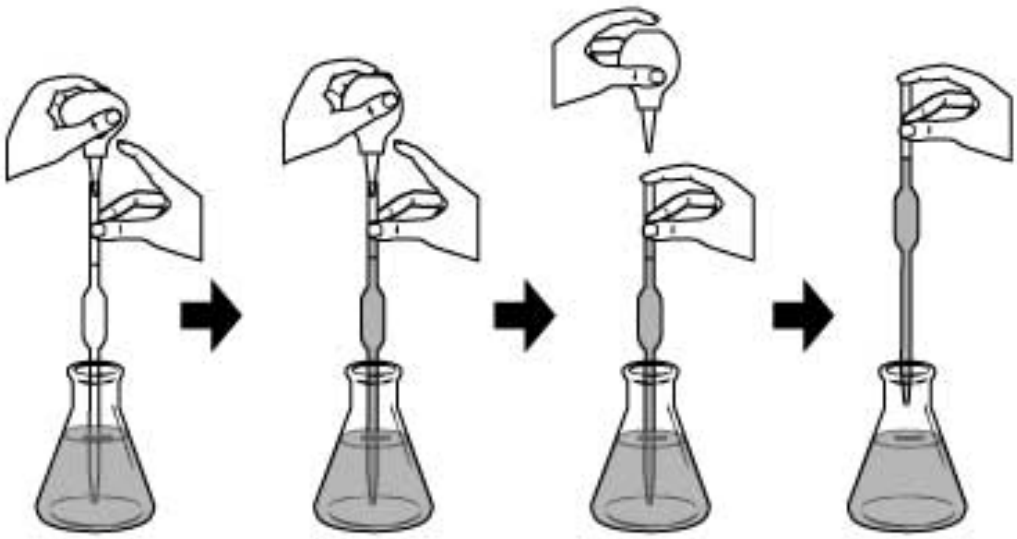
c. To condition a volumetric pipet, draw a small amount of your working solution into the pipet just above the volume mark. Drain the solution into an appropriate waste container.

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.

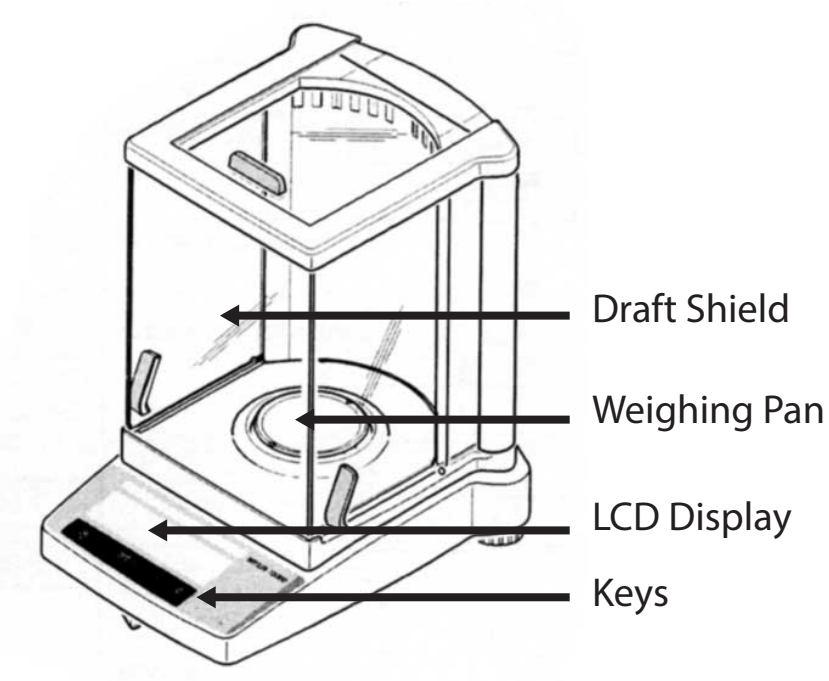


Figure 5. The Analytical Balance

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.

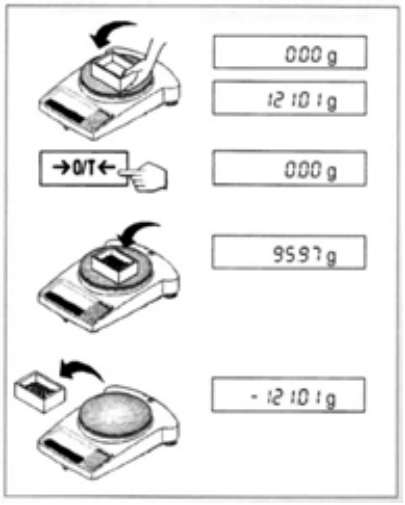


Figure 6. Taring

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.

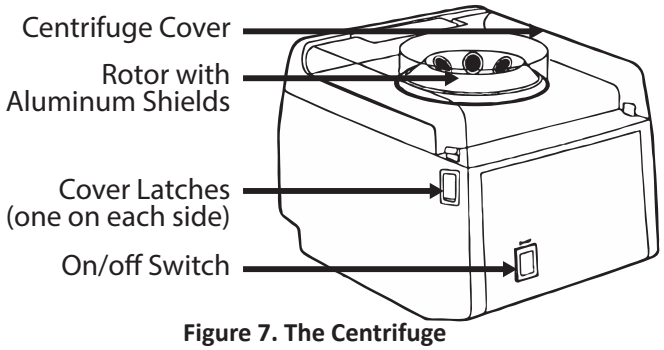


Figure 7. The Centrifuge

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.

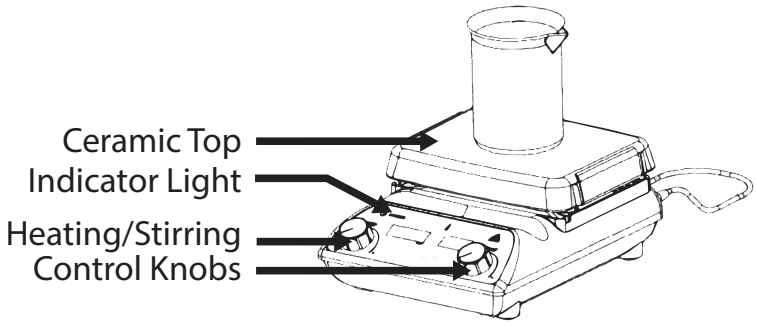


Figure 8. The Hot Plate/Stirrer

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.

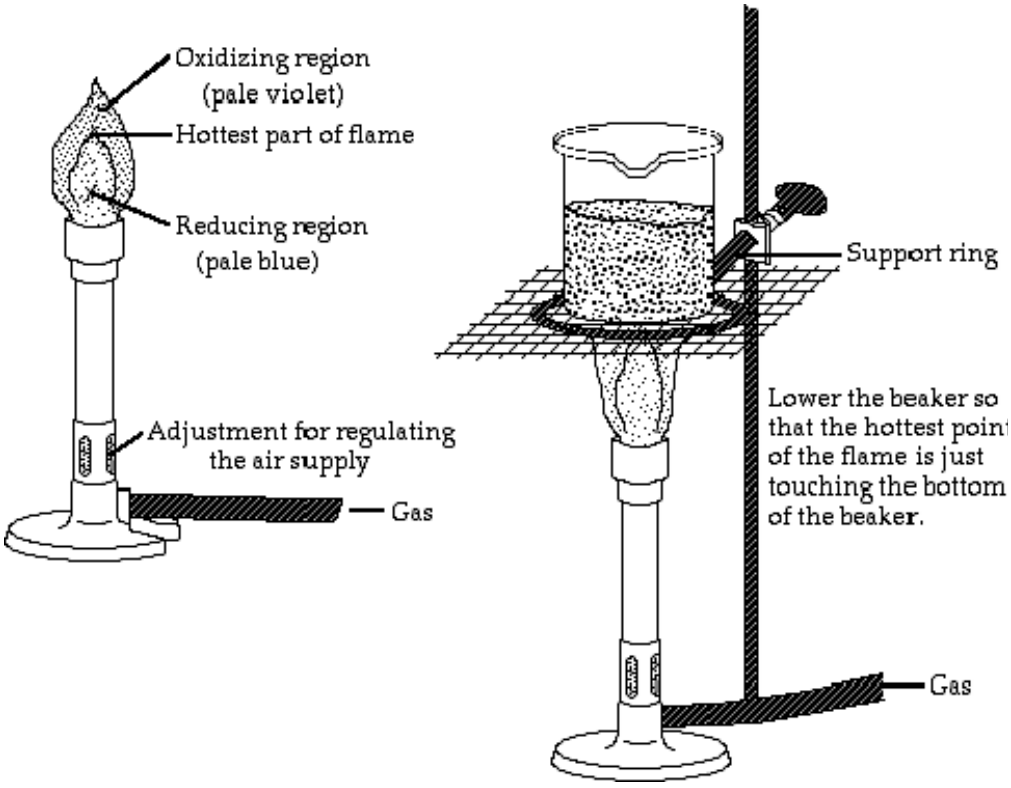


Figure 9. The Bunsen Burner

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!

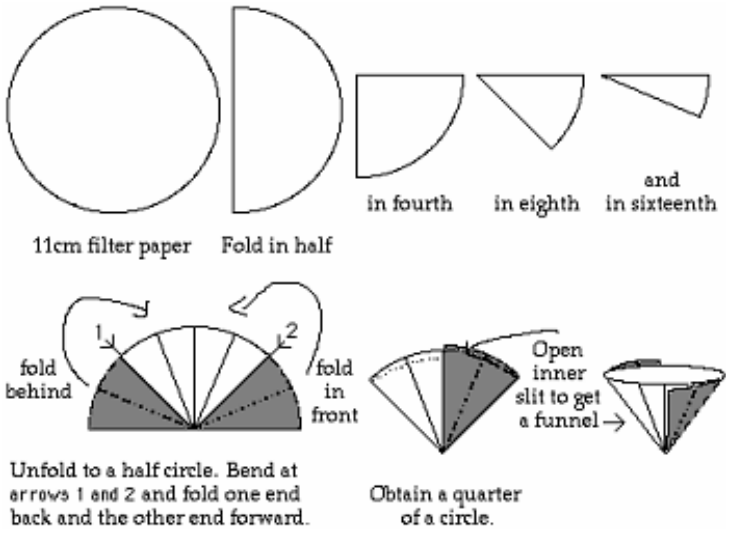
Filtration

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

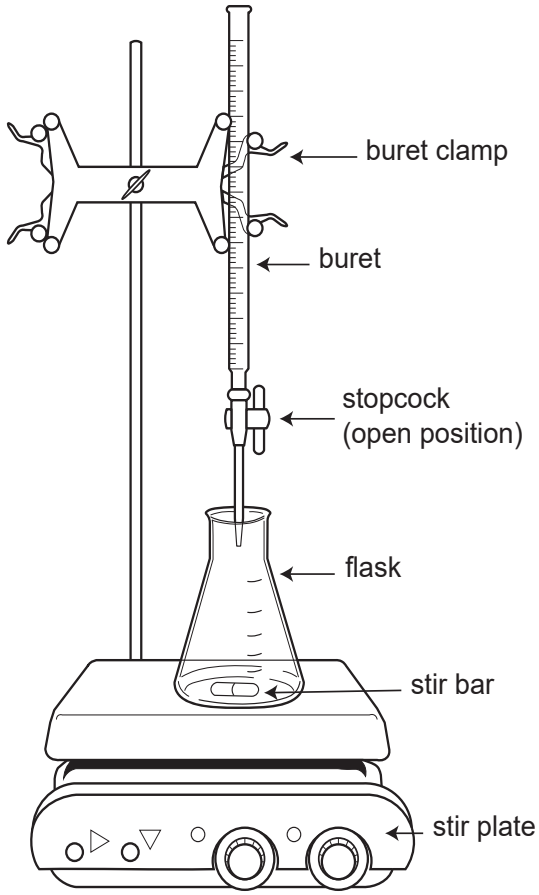


Figure 10. Titration Setup

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.

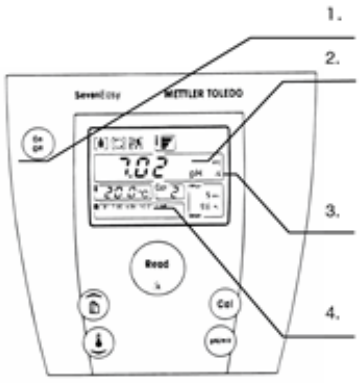
4. Add your stir bar, solution, and indicator to the titration flask, and place the flask underneath the tip of the buret. Turn on the stirrer and slowly increase the stirring speed.

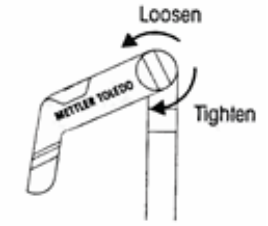
5. Lower the buret tip into flask without touching the flask sides. You are now ready to titrate!

pH Meter Operating Instructions

1. Preparing the pH meter

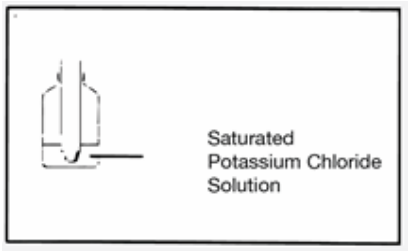
1. Turn on the pH meter.
2. Meter must be in pH mode. If in mV mode, press the **pH/mV** button.
3. Make sure pH meter is showing /Ā. If not shown, press and hold **Read** button for 2 seconds.
4. Lower left window must show **B1 7.00 4.00 10.01 1.68**. If not, ask your TA to adjust the setting.
5. You 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.**

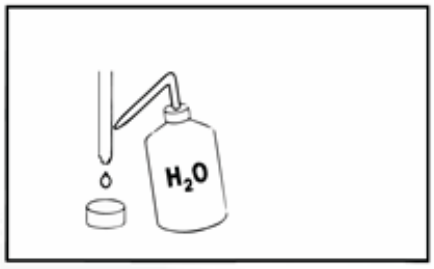


2. Calibrating the pH meter

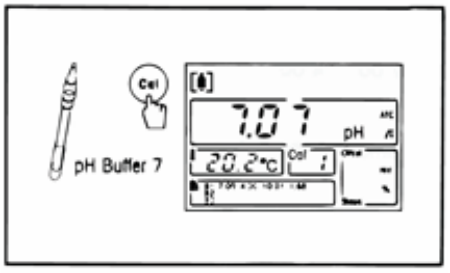
Note: You only need to calibrate the pH meter **once** per lab period.

1. Rinse the electrode with DI water.
2. Blot dry with Kimwipe.

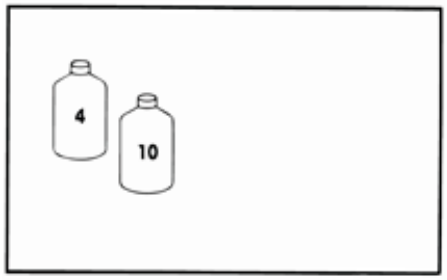
WARNING: Do **NOT** rub the electrode with Kimwipe. Rubbing the electrode may build up static charge and damage the electrode.



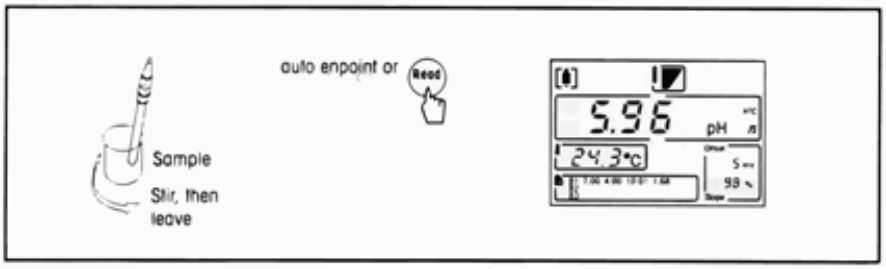
3. Place electrode in pH 7 buffer standard (yellow).
4. Press the **Cal** button.
5. Wait for the display to stop blinking.



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

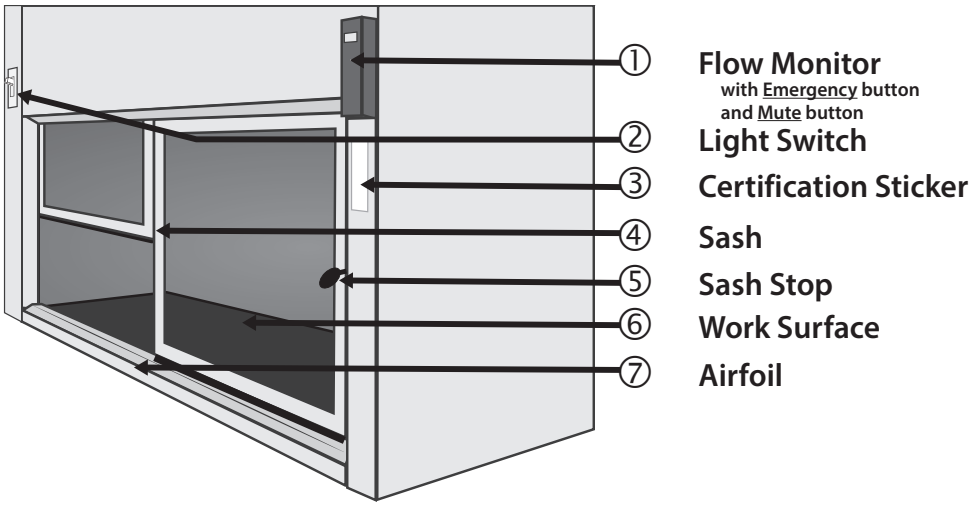


1. After calibration, place the electrode in sample solution and press **Read**.
2. Wait for the reading to stabilize.

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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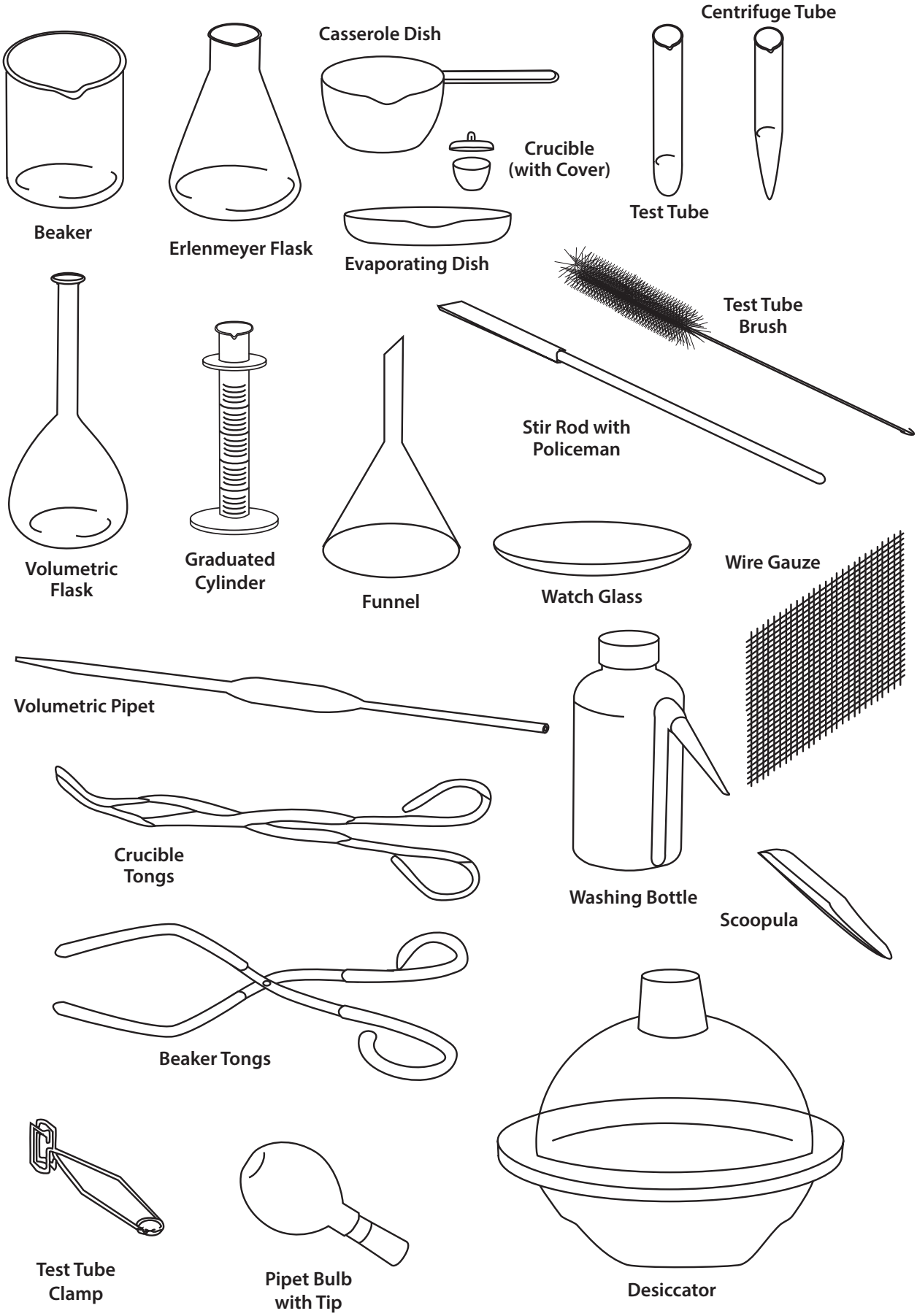
Beaker
Erlenmeyer Flask
Casserole Dish
Crucible
(with Cover)
Evaporating Dish
Centrifuge Tube
Test Tube
Test Tube
Brush
Stir Rod with
Policeman
Volumetric
Flask
Graduated
Cylinder
Funnel
Watch Glass
Wire Gauze
Volumetric Pipet
Crucible
Tongs
Washing Bottle
Scoopula
Beaker Tongs
Test Tube
Clamp
Pipet 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, SLB 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, SLB 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]