

Chemistry 2A

Laboratory Manual

Standard Operating Procedures



Department of Chemistry
University of California - Davis
Davis, CA 95616
Summer 2015

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 _____

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.

Acknowledgements

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. In addition, many undergraduates have been involved in the development of experiments as part of undergraduate research projects.

HAZARD CLASS 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 designate this laboratory as a use area:

Carcinogen Use Area

Corrosives Use Area

Metal Powders Use Area

Reproductive Hazards Use Area

Water Reactives Use Area

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Introduction

Experiment List

Below is an indication of the order 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

Introductory Laboratory Techniques

Nomenclature Lab

Observing Chemical Reactions

Reactions of Copper

General Analytical Techniques

Volumetric Analysis (Part I and II)

Spectrophotometry (Part I and II)

Determination of Avogadro's Number

***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 Policies

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. 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. Goggles must be worn by EVERY person in the lab until EVERYONE has finished with the experimental procedure and has put away ALL glassware. Safety goggles may not be modified in any manner.
3. Closed-toe, closed-heel shoes that cover the entirety of the foot must be worn at all times.
4. Clothing (baggy sleeves and pant legs are NOT allowed) that completely covers your arms and legs must be worn at all times in the laboratory (long skirts, tights, or leggings do NOT qualify). Inadequate protection often leads to injury. Avoid wearing expensive clothing to lab as it may get damaged.
5. Lab Coats of 100% cotton are REQUIRED upon entering lab.
6. Absolutely NO food or drinks are allowed in the laboratory. This prohibition applies to the storage of food and the consumption of food, beverages, medicines, tobacco, and chewing gum. Contact lenses and cosmetics are not to be applied while in the laboratory. Infractions will result in expulsion from the laboratory. Because cell phones or other personal electronic media can easily be damaged in the laboratory, use of such devices is at the student's own risk.
7. Learn the location and how to operate the nearest eyewash fountain, safety shower, fire extinguisher, and fire alarm box. First aid for acid or base in the eyes is to wash with copious

Introduction

SAFETY RULES FOR TEACHING LABORATORIES

amounts of water using the eyewash fountain for 15 minutes; then go immediately to the Student Health Center for further treatment. First aid for acid or base on skin or clothing is to wash thoroughly with water for 15 minutes. Use the emergency shower if appropriate, removing contaminated clothing for thorough washing. If the safety shower or eyewash is activated, the exposed person must be accompanied to the Student Health Center for further evaluation.

8. All operations in which noxious or poisonous gases or vapors are used or produced must be carried out in the fume hood.

9. Confine long hair while in the laboratory. Hair can catch on fire while using open flames.

10. Mouth suction must never be used to fill pipets. Always use a bulb to fill pipets.

11. All accidents, injuries, explosions, or fires must be reported at once to the LI. In case of serious injury, the LI or Lab Supervisor must call 911 for an ambulance. In cases where the LI and Lab Supervisor decide the extent of an injury warrants evaluation/treatment, the student must be accompanied to the Student Health Center. Students are also encouraged to seek medical attention if the student deems it necessary. The student must always be accompanied to the Student Health Center.

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

13. Keep your working area clean – immediately clean up ALL spills or broken glassware. Exercise appropriate care to protect yourself from skin contact with all substances in the laboratory. Clean off your lab workbench before leaving the laboratory. Skateboards, rollerblades, and other such personal equipment must be stored outside of the laboratory. Personal electronics are only permitted when needed for the laboratory.

14. Put all toxic or flammable waste into the appropriate waste container(s) provided in your laboratory.

15. 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 CAPPED before you take them into the hallway to the dispensary. Never take uncapped glassware containing chemicals into the hallways or other public areas.

16. Laboratory doors must remain closed except when individuals are actively entering or exiting the lab. DO NOT prop the door open with chairs, stools, or any other objects.

17. The student must have at least ONE UNGLOVED HAND when outside the laboratory. Only use the ungloved hand to open doors. Gloves are presumed to be contaminated and must not come into contact with anything outside the laboratory except chemical containers.

18. Specific permission from your LI is required before you may work in any laboratory 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.

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

20. 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 that 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 dress code.

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

Experiments

Introductory Laboratory Techniques

INTRODUCTION

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

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

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

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

This lab is designed to 1) acquaint you with the equipment in your locker, and 2) introduce you to some basic laboratory techniques. A word of warning: a few of you may find this and other beginning laboratories in Chemistry 2A to be somewhat tedious, especially if you've had a good high school chemistry laboratory course. However, please be patient as the goal is to give all students a good common background so every student has an excellent chance of success with the later, more difficult experiments. Remember that as pre-laboratory preparation, you should come to the laboratory with Title, Purpose, Procedure, and Data Tables entered in your notebook. At the end of the laboratory period you should have your TA sign and date your laboratory notebook near your data tables. At the next laboratory meeting you will submit a completed report.

Introductory Laboratory Techniques

PROCEDURE

Common Laboratory Procedures

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

In these procedures you will learn to properly:

- a) use a balance,
- b) measure the volume of a liquid, and
- c) use a Bunsen burner.

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

Safety: Wear your PPE. Use a bulb with an Eppendorf tip to fill a pipet.

PROCEDURE

You will work in pairs on this experiment. The actual data analyses and the written reports must be done entirely independently of your lab partner or other students. Make sure that you avoid unauthorized collaboration and plagiarism. All suspected violations of the Code of Academic Conduct will be referred to Student Judicial Affairs.

Stock Chemicals Used

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

Part I. Measuring Volumes

A. Using a Pipet to Measure Volume

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

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

2. Confirm that your 10.00 mL volumetric pipet is clean by filling to above the mark with deionized water and then letting it drain. Your pipet is a transfer pipet that is calibrated “to deliver” (TD) rather than “to contain” (TC). The last drop of liquid should not drain out of the tip of a TD pipet in normal use. However, there should

be no water drops left on the side walls. The presence of such drops indicates that your pipet is dirty.

Helpful hint: You will use your pipet in many of the experiments in Chemistry 2. It is important that you always clean it at the end of the day, and rinse it thoroughly with deionized water before storage. Pipet cleaning solution is located in a 1 L bottle at the front of the room. Follow the instructions on the label. Remember that your pipet is calibrated to deliver.

3. Measure and record the mass of a clean 125 mL Erlenmeyer flask.
4. Measure and record the temperature of the room and of the water that was set aside in step 1. The two temperatures should agree before you continue. Read the thermometer to the closest one tenth of a degree, using your best estimate. Please be especially careful with the thermometer.
5. Use your pipet to deliver 10.00 mL of the equilibrated water into the Erlenmeyer flask. Note the precision used here.
6. Measure and record the mass of the flask and the water.
7. Repeat steps 5 and 6 at least two additional times without emptying out your flask between trials.
8. Calculate the mass of water delivered by your pipet for each trial. Use your mass of water and the volume of the volumetric pipet to calculate the density of water for each trial. Calculate the average density, standard deviation, and the 90% confidence limits for the average density.
9. Use the temperature of your water along with the values of mass and volume of water given in Table I to calculate the accepted values for the density of water.

Introductory Laboratory Techniques

PROCEDURE

10. Determine the relative error with respect to the average density of water.

The relative error is defined by:

$$\text{relative error} = \frac{|\text{experimental result} - \text{accepted value}|}{\text{accepted value}} \times 100$$

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

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

Table I gives the corrected volume in mL occupied by 1.0000 g of water when weighed in air against stainless steel weights for different temperatures. Two effects are included in this volume per 1.0000 g; first, the change in the density of water with temperature; and second, a much smaller correction due to buoyancy. The buoyancy correction arises since the balance was set to zero with a certain mass of air on the balance pan. The volume of water displaces some of this air from the balance pan that makes the water appear lighter than it really is. The contribution of buoyancy to the results in Table I is roughly 0.0011 mL per 1.0000 g of water.

B. Using a Buret to Measure Volume

1. Discard the water in your Erlenmeyer flask, and re-measure the mass of the flask. The inside of the flask need not be completely dry because any water left in it is from the previous procedure and is at the same temperature as the new water you will be adding.
2. Use a 25 mL buret and **accurately** measure out *about* 12 mL of room temperature deionized water from part A into the flask. You should read the buret to the closest one hundredth mL (e.g., 12.14 mL). In your laboratory notebook, record your initial buret reading and your final buret reading. The volume of water delivered by the buret is the difference between the final and initial buret reading.
3. Measure and record the mass of the flask and the water.

4. Repeat steps 2 and 3 at least two additional times without emptying out your flask between trials.
5. Calculate the mass of water delivered by your buret for each trial. Use your mass of water and the volume of the water delivered by your buret to calculate the density of water for each trial. Calculate the average density, standard deviation, and the 90% confidence limits for the average density.
6. Assuming that the water temperature has not changed, compare your experimental value of the density of water to the accepted value of the density of water you calculated in part A.
7. Determine the relative error with respect to the average density of water when measured by the buret.

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

C. Using a Beaker to Measure Volume

1. Measure and record the mass of clean and dry 100 or 150 mL beaker. Note this beaker needs to have a 50 mL graduation mark.
2. Use your clean and dry 100 or 150 mL beaker and carefully measure out 50 mL of your room temperature water.
3. Measure and record the mass of the beaker and the water.
4. Empty out your beaker and carefully measure out another 50 mL of your room temperature water. There is no need to reweigh the empty beaker.
5. Measure and record the mass of the beaker and water.
6. Repeat steps 4 and 5 at least once more
7. Use your mass of water and the volume of the water measured by your beaker to calculate the density of water for each trial. Calculate the average density, standard deviation, and the 90% confidence limits for the average density.
8. Assuming that the water temperature has not changed, compare your experimental value of the density of water to the accepted value of the density of water you calculated in part A.
9. Determine the relative error with respect to the average density of water when measured by the flask.

Introductory Laboratory Techniques

PROCEDURE

Part II. Drying a Hydrate

1. As illustrated by the TA, place a clean crucible on a wire triangle above a Bunsen burner. With the TA watching, light the Bunsen burner and adjust the flame and the height of the wire triangle so that the crucible is positioned in the hottest part of the flame.
2. Heat the crucible for five minutes to make sure it is dry, and then remove it from the wire triangle using crucible tongs and place it on your desktop to cool.
3. After the crucible has returned to room temperature (approximately five minutes), measure and record its mass to the thousandth of a gram (milligram).
4. Weigh into your crucible 1.0 - 1.2 g of manganese(II) sulfate monohydrate, $\text{MnSO}_4 \cdot \text{H}_2\text{O}$, recording the exact mass to the thousandth of a gram (milligram).
5. Heat the crucible with its contents for five minutes, and then remove it to your desktop using crucible tongs.
6. After the crucible and its contents have returned to room temperature, measure and record the mass.
7. Repeat steps 5 and 6 until the mass readings are consistent. (Mass no longer decreases after heating.)
8. Calculate the mass loss by your sample upon heating.
9. Transfer the contents of your crucible to the waste container located in the fume hood.

Clean-up: Solid dry manganese(II) sulfate may be disposed of in the proper waste container found in the fume hood. Clean your volumetric pipet and buret with deionized water only. All other glassware may be cleaned with tap water and rinsed with deionized water. Always, let your glassware air-dry. If time permits, now would be a good time to also 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 rinsed with water using a sponge. Inform your TA that you are leaving and be sure that your laboratory drawer gets locked.

DATA ANALYSIS

Part IA. Measuring Volumes with a Pipet

For each of your three acceptable trials, what was the measured mass of water delivered by your 10.00 mL pipet?

For each of your three acceptable trials, what was the density of water that you determined with your 10.00 mL pipet?

Using the data from your three trials with the 10.00 mL pipet, what is the average, standard deviation, and 90% confidence limits of the average?

What was the temperature of the room and the temperature of the water to the closest degree Celsius when you made your measurements for this laboratory?

Referring to Table I in the laboratory manual, what is the accepted value for the volume of 1 gram of water at the temperature you reported in question 4?

Using the appropriate volume of 1 gram of water from Table I, what is the accepted value for the density of water at the temperature of your measurements?

Using your average value for the density of water determined with your 10.00 mL pipet and the accepted value for the density of water, what is the relative error in the density that you determined with your 10.00 mL pipet?

Part IB. Measuring Volumes with a Buret

For each of your three acceptable trials, what was the volume of water that you measured with your buret?

What was the mass you determined for each corresponding volume that you measured with your buret?

For each of your three acceptable trials, what is the density of water that you determined with your buret?

Using the data from your three trials using the buret, what is the average, standard deviation and 90% confidence limits of the average.

Using your average value for the density of water determined with your buret and the accepted value for the density of water that you found in question 6, what is the relative error in the density that you determined with your buret?

Introductory Laboratory Techniques
DATA ANALYSIS

Part IC. Measuring Volumes with a Beaker

For each of your three acceptable trials, what was the volume of water that you measured with your beaker?

What was the mass of water that you determined for each corresponding volume that you measured with your beaker?

For each of your three acceptable trials, what is the density of water that you determined with your beaker?

Using the data from your three trials using the beaker, what are the average, standard deviation and 90% confidence limits of the average?

Using your average value for the density of water determined with your beaker and the accepted value for the density of water that you found in question 6, what is the relative error in the density that you determined with your beaker?

If you wanted to accurately measure a 20.00 ± 0.02 mL volume of a liquid, which piece of glassware would you use; 10.00 mL pipet, buret, or Erlenmeyer Flask? If you wanted to quickly measure about 20 mL, which would you use?

Generally, a pipet is more accurate than a buret. Do your experimental results support this statement? If yes, explain. Explain why a student's experimental results may not support the statement?

Part II. Drying a Hydrate

What was the mass of your empty crucible?

What was the mass of your crucible + $\text{MnSO}_4 \cdot \text{H}_2\text{O}$?

What was the initial mass of your sample of $\text{MnSO}_4 \cdot \text{H}_2\text{O}$?

What was the final mass of your crucible + sample after you had heated it to constant weight?

What was the final mass of your sample?

What was the mass lost by your sample upon heating?

What is the chemical formula of the final product?

What is the theoretical percent mass of water in manganese(II) sulfate monohydrate?

Calculate your experimentally determined percent mass of water in manganese(II) sulfate monohydrate.

What is your relative error with respect to percent mass of water in the monohydrated sample?

Why did your empty crucible need to be dried by heating? If your crucible had not been dried before heating the sample how would that have affected your calculations in Question 28?

Nomenclature Lab

INTRODUCTION

Whenever you begin study of a new field, what do you have to do first? You have to learn the language of that field. Every field of study has its own vocabulary. Thus, before you can communicate you have to learn the words that people use in that field, or you will have a very hard time learning the concepts that are being taught. This is certainly also true in chemistry. In chemistry we use words that you may or may not have heard before such as *electrolysis*, *effusion*, *hybridization*, *resonance*, and *stoichiometry*. Chemists also use words that have a different meaning than the common definition such as the word *mole*. Therefore, a good grasp of the language of chemistry is critical to your success in a chemistry course. However, studying a new language is difficult and not considered enjoyable by many people. Luckily, when instructors teach chemistry they generally try to use the new words only when they need them making the learning of the language much easier.

However, there is one part of the chemical vocabulary that simply can't be put off, and that is the language of chemical nomenclature. When instructors talk about the properties of chemicals (which is what chemistry is!), it is critical that you recognize the chemical that is being discussed. For example, if during the discussion of chemical reactions an instructor mentions that potassium chlorate is heated to produce potassium chloride and oxygen, it is critical that you are able to write the chemical reaction for this process. Thus you must learn chemical nomenclature as soon as possible. As further impetus to your study keep in mind that studies show a good correlation between learning nomenclature and final course grade. That is, students who learn nomenclature early and well have greater success in the course.

How do you learn chemical nomenclature? You practice and practice and practice. The following pages contain notes that have been prepared as an aid in your pre-workshop preparation. Please read these over carefully before coming to the laboratory.

ASSIGNMENT

View the **On-line Prelab Presentation of Nomenclature**. It is a detailed presentation of the rules of nomenclature. Examples are given in the presentation, as well as, practice problems.

Complete the 50 point/50 question on-line quiz. You can find it under "Post-Laboratory" in the on-line program. Unlike the prelab quizzes, you can log out of the quiz at any time without penalty and continue the quiz later. The on-line program times out at 20 minutes. Also, unless you click on the submit button for the particular question you are working on, the program will not count the question against you. For example, if you are working on question 10 when the program times out or you log out, the program will begin at question 10 the next time you log-in to continue the Nomenclature quiz.

Nomenclature Lab
ASSIGNMENT

I. IONIC COMPOUNDS

A. Cations

1. Monatomic

a) Monatomic cations are most commonly formed from metallic elements. They take the name of the element itself:

Na^+	sodium ion	Zn^{2+}	zinc ion
---------------	------------	------------------	----------

b) If an element can form more than one positive ion, the positive charge of the ion is indicated by following the name of the metal with a Roman numeral in parenthesis:

Fe^{2+}	iron(II) ion	Cu^+	copper(I) ion
Fe^{3+}	iron(III) ion	Cu^{2+}	copper(II) ion

Experience is required to know which elements commonly exist in more than one charge state. An older method still widely used for distinguishing between two differently charged ions of a metal uses the endings -ous or -ic; these endings represent the lower and higher charged ions, respectively. They are used together with the root of the Latin name of the element:

Fe^{2+}	ferrous ion	Cu^+	cuprous ion
Fe^{3+}	ferric ion	Cu^{2+}	cupric ion

2. Polyatomic

a) The only common polyatomic cations are those given below:

NH_4^+	ammonium ion	Hg_2^{2+}	mercury(I) or mercurous ion
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The name mercury(I) ion is given to Hg_2^{2+} because it can be considered to consist of two Hg^+ ions. Mercury also occurs as the monatomic Hg^{2+} ion, which is known as the mercury(II) or mercuric ion.

B. Anions

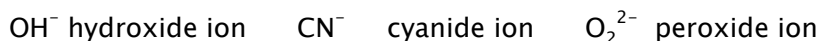
1. Monatomic

Negative ions are called anions. Monatomic anions are most commonly formed from atoms of the nonmetallic elements. They are named by dropping the ending of the name of the element and adding the ending -ide:

H^-	hydride ion	O^{2-}	oxide ion	N^{3-}	nitride ion
F^-	fluoride ion	S^{2-}	sulfide ion	P^{3-}	phosphide ion

2. Polyatomic

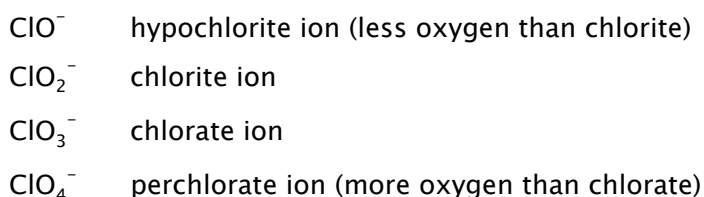
a) Only a few common polyatomic ions end in -ide:



b) There are many polyatomic anions containing oxygen. Anions of this kind are referred to as oxyanions. A particular element, such as sulfur, may form more than one oxyanion. When this occurs, rules for indicating the relative numbers of oxygen atoms in the anion are used. When an element has two oxyanions, the name of the one that contains more oxygen ends in -ate; the name of the one with less oxygen ends in -ite:

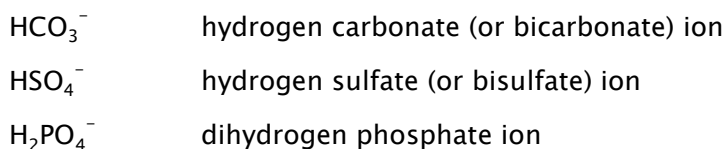


When the series of anions of a given element extends to three or four members, as with the oxyanions of the halogens, prefixes are also employed. The prefix hypo- indicates less oxygen, whereas the prefix per- indicates more oxygen:



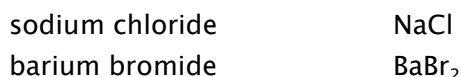
Notice that if these rules are memorized, only the name for one oxyanion in a series is needed to deduce the names of the other members.

c) Many polyatomic anions that have high charges readily add one or more hydrogen ions to form anions of lower charge. These ions are named by prefixing the word hydrogen or dihydrogen as appropriate, to the name of the hydrogen-free anion. An older method which is still used, is to use the prefix bi-:



C. Naming Ionic Compounds

The names of cations and anions are combined to name and write the formulas for ionic compounds:



Nomenclature Lab
ASSIGNMENT

copper(II) nitrate	$\text{Cu}(\text{NO}_3)_2$
mercurous chloride	Hg_2Cl_2
aluminum oxide	Al_2O_3

The overall zero charge is provided for by adjusting the ratios of cations and anions. In the second example, two Br^- anions are required to balance the charge of the single Ba^{2+} cation. In the third example, two nitrate ions, NO_3^- , are required to balance the charge of the Cu^{2+} ion. Notice that the formula for the entire anion must be enclosed in parentheses so that it is clear that the subscript 2 applies to all the atoms of the anion. The final example, aluminum oxide, is a little more complicated in that more than one of both cation and anion are needed to achieve charge balance. Two Al^{3+} cations are needed to balance the total charge of three O^{2-} anions.

II. ACIDS

This important class of compounds are named in a special way. For purposes of naming, the acids may be thought of as formed from hydrogen ions and an anion. When the anion is a simple monatomic species, the name of the acid has a prefix hydro- and an ending, -ic, as in these examples:

chloride ion (Cl^-)	hydrochloric acid (HCl)
sulfide ion (S^{2-})	hydrosulfuric acid (H_2S)

Many of the most important acids are derived from oxyanions. The name of the acid is related to the name of the anion; when the name of the anion ends in -ate, the name of the acid ends in -ic. Anions whose names end in -ite have associated acids whose names end in -ous. Prefixes in the name of the anion are retained in the name of the acid. These rules are illustrated by the oxyacids of chlorine:

hypochlorite ion (ClO^-)	hypochlorous acid (HClO)
chlorite ion (ClO_2^-)	chlorous acid (HClO_2)
chlorate ion (ClO_3^-)	chloric acid (HClO_3)
perchlorate ion (ClO_4^-)	perchloric acid (HClO_4)

(Continued on next page)

III. NON-IONIC COMPOUNDS

Before we end this discussion, we will consider the systematic names of compounds that are nonionic. These are named with the more electropositive charged element first. The relative number of atoms of each element is indicated by prefixes; for example, mono-, di-, tri-, tetra-, penta-, and hexa-, which mean 1, 2, 3, 4, 5, and 6, respectively. We use the same suffixes in naming the more negative element as is used in naming ionic compounds. Some examples illustrate the rules:

NF ₃	nitrogen trifluoride	CO ₂	carbon dioxide
N ₂ F ₄	dinitrogen tetrafluoride	SO ₂	sulfur dioxide
CO	carbon monoxide	SO ₃	sulfur trioxide
CCl ₄	carbon tetrachloride	P ₂ O ₅	diphosphorous pentoxide
CHCl ₃	chloroform (trichloromethane)	NH ₃	ammonia (nitrogen trihydride)

The other rules for naming compounds will be used as the need arises. We will also frequently encounter common names of compounds that are still widely used. These compounds were discovered a long time ago and are commonly used in the applied sciences. The common names of these chemicals must simply be memorized.

Observing Chemical Reactions

INTRODUCTION

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

How do you know that mixing two chemicals results in a chemical reaction? Look for as many physical indications as possible. Does the color of the solution change? Does it heat up? Does it cool down? Is gas evolved? Use all of your senses- except smell and taste; remember, don't smell or eat the chemicals! It cannot be emphasized enough that making good observations, and writing them down, is critical to successful investigations in science. Think about how often you have said to yourself, "I'll remember the phone number until I get home," and then promptly forgotten it. It is much easier to forget something you have noted about a new chemical reaction, especially something you did not realize was significant at the time, than something you considered important in the first place. If you note a change or a lack of change, write it down!

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

In preparation for the Volumetric experiment, each pair must obtain about 3.0 grams of primary-standard grade potassium acid phthalate, KHP, in a vial and dry it in an oven at 110° C for 2 hours. Place the vial of KHP in a small, beaker to keep it from spilling. Label your beaker using a graphite pencil in the white frosted area. Cover the beaker with a watch glass and place it in the oven. After two hours, remove your beaker from the oven. While keeping the watch glass on top of the beaker, let it cool until it is warm but safe to handle. Remove the watch glass and place the beaker containing the uncapped vial in a desiccator.

Observing Chemical Reactions

PROCEDURE

Warning

Use care when handling acids and bases. Silver nitrate will stain clothing and skin; wear gloves. Dispose of solutions containing silver in the waste jars in the hood. Wear your goggles.

PROCEDURE

You will work in pairs on this experiment. Each student will submit a separate post-laboratory report.

Stock Chemicals Used

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

Part I. Reactions of the Metal Salt Solutions

In this part of the experiment you will experiment with four metal salts and five other reagents. The metal salts you will use are silver nitrate, strontium nitrate, magnesium nitrate and aluminum nitrate. The other reagents you will work with are 6 M hydrochloric acid, 3 M sulfuric acid, 6 M ammonium hydroxide, 6 M sodium hydroxide and 6 M nitric acid. The salt solutions will be found on the chemical shelves, and the acids and bases will be found on the trays by your lab station. If the reagent present in the laboratory is more concentrated than this, dilute it.

React each reagent with the individual metal salts

1. Using a disposable transfer pipet, transfer approximately 1 mL of a 0.1M metal salt solution to a clean test tube.
2. Using the dedicated transfer pipet attached to the reagent bottles, slowly add a couple of drops of one of the reagents to one of the metal salts. Record your observations.
3. Add a couple more drops and record your observations. Continue until you are sure that you have added an excess of the reagent. It will not take more than 0.5 mL to reach an excess of the reagents.

4. There are twenty possible combinations of salts with reagents. Try them all. You may also want to see what happens if you add more than one reagent to the salt solution.

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

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

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

Part II. Analyzing an Unknown

In this part of the experiment you will use the data you have accumulated to tell which of the salts are present in an unknown. Your unknown contains two of the four metal salts you have worked with. Develop a procedure that will distinguish between these four compounds and use it to identify the composition of your unknown. You may need to be able to separate a solid precipitate from a solution. Instructions for separation follow.

1. Transfer the solution and the precipitate to a centrifuge test tube. Fill a second test tube with water until the volumes in the two test tubes are approximately the same.
2. Place the two test tubes in the centrifuge. The test tubes should be placed opposite each other so that their weight is balanced as the centrifuge spins. As there are likely to be more people using the centrifuge, make sure that your test tubes are labeled so that you can identify them when the centrifuge stops.
3. Turn on the centrifuge and allow it to spin for a minute. When the centrifuge stops spinning remove your test tube carefully- you do not want to disturb the solid at the bottom of the tube.
4. If you are trying to completely remove a salt from a solution, add a little more of the reagent that caused the precipitation. If more solid forms in the solution, recentrifuge and repeat this step.
5. Decant the supernatant solution from the solid and reserve. Avoid disturbing the precipitate when pouring off the solution.
6. Add a few milliliters of deionized water to the precipitate and stir. This washes any excess reagent away from the precipitate. Recentrifuge. Decant the supernatant and combine with the reserved solution from Step 5. You have now completed the separation of your precipitate from solution. Separate tests may be performed (as needed) on the precipitate or the supernatant solution.

Question C: Why do you test for complete precipitation in Step 4 if you are going to do any further chemical tests on the supernatant?

Observing Chemical Reactions

DATA ANALYSIS

Clean up: Pour any solutions or solids containing silver or aluminum into the Cation Metal Waste jar in the fume hood. Pour any other solutions down the drain

DATA ANALYSIS

Why must you use de-ionized water rather than tap water in performing the reactions and any required dilutions?

Which metal salts formed a precipitate when a few drops of 6M hydrochloric acid were added?

What is the correct chemical equation for the reaction of strontium nitrate with sulfuric acid?

What chemical equation represents what occurs when ammonium hydroxide is added to a solution of silver nitrate?

What chemical equation properly describes the reaction of magnesium nitrate solution with added sodium hydroxide?

What chemical equation properly describes the reaction of aluminum nitrate solution with added ammonium hydroxide?

Why is it necessary to test for complete precipitation if a supernatant solution is going to be subsequently tested for ions?

What is the letter of the unknown solution that you analyzed?

What metal salt solutions have you identified in your unknown solution?

Provide a cohesive summary of the reactions you undertook and the observations you made in this laboratory session. If you did not obtain a perfect score on your unknown, explain what error in procedure or observation you might have made that resulted in a false conclusion.

Reactions of Copper

INTRODUCTION

In this experiment you will do a series of reactions with the element copper. These reactions will involve the use of some new techniques and some interesting color changes. In addition, they will illustrate some of the classes of chemical reactions that occur in nature, for example, *oxidation-reduction reaction*. It is important that you make careful observations as you carry out these procedures. It is also very important that you do the questions as they appear in the procedures. This will keep you from falling into the trap of the cookbook approach that was discussed earlier and help you more fully understand the principles involved in the experiment.

In the first reaction you will dissolve elemental copper and make a solution of copper nitrate. This solution will then be treated with a base that causes a precipitation. The resulting precipitate will then be heated to produce an oxide of copper. The oxide will then be treated with sulfuric acid to produce a blue colored solution of copper(II) sulfate. Finally, the copper ion will be reduced by zinc to produce copper metal that you will collect and dry.

In some steps of this experiment you are given amounts of chemicals to use so that you have an idea of the scale at which you should work. In other steps you are left to make your own decisions. Keep in mind the things you learned about chemical reactions in the last experiment. They will be of use to you here.

Reactions of Copper

PROCEDURE

PROCEDURE

You will work in pairs on this experiment. Each student will submit a separate post-laboratory report.

Stock Chemicals Used

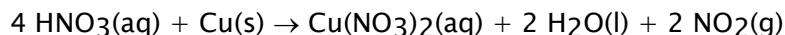
Chemical	Maximum Amount Used
6M Hydrochloric Acid	Small Amount
6M Nitric Acid	Small Amount
3M Sulfuric Acid	Small Amount
6M Sodium Hydroxide	10–20 mL
Copper Wire	0.5 g
Granular Zinc, 20 mesh	~ 1 g
Conc. Nitric Acid (15M)	~4 mL

Safety: Carry out all steps that produce fumes in the hoods as described. Be especially careful with acids and bases. Wear your goggles.

1. Making a Solution of Copper(II) Nitrate:

Accurately weigh out 0.500 - 0.530 g sample of copper wire and record the measurement in your notebook. Using your pencil or pen, coil the wire into a flat spiral and place it into a clean 250 mL Erlenmeyer flask. Measure 4 mL of concentrated nitric acid found in the FUMEHOOD into a graduated cylinder. Keep the flask and graduated cylinder in the hood and pour the nitric acid into the flask. Make observations regarding any changes that occur during the reaction. **Be careful not to breathe any of the fumes!** Make notes of your observations in your notebook. Swirl the flask to be sure all the copper dissolves. If all the copper fails to dissolve within 10 minutes, then add an additional mL of nitric acid. Once all the copper has dissolved then add about 100 mL of deionized water, which is not usual but needed for this particular step, and take the flask to your laboratory bench. **Be sure that no more fumes are being created before you remove the flask from the fume hood.**

Question A: This reaction is an oxidation–reduction reaction that is somewhat complicated.



Identify the elements oxidized and reduced in this reaction and indicate how many moles of electrons are transferred between the elements being oxidized and reduced.

2. Synthesis of solid copper(II) hydroxide:

The second reaction that you should perform is the synthesis of solid copper(II) hydroxide from the copper(II) nitrate you made in the last step. This may be accomplished by adding 6 M sodium hydroxide to the copper solution. Add a

minimum of sodium hydroxide, but also be sure that you have added enough to complete the reaction. This volume will be around 10 - 20 mL.

Question B: How do you determine that you have added enough sodium hydroxide?

Question C: Write the balanced chemical equation for the reaction in this step.

3. Formation of the Oxide:

To change the hydroxide to the oxide, slowly and carefully heat the solution containing the precipitate over a hot plate. **Be sure to stir the solution continuously during the heating with a glass stir rod.** This will avoid the possibility of "bumping" which can occur when a large steam bubble forms within a solution due to a local region of overheating. Bumping will cause a loss of product in addition to possible injury! Watch the solution carefully and record observations. When the reaction is complete, remove the hot plate and continue to stir for a couple of minutes to avoid bumping of the cooling solution. Then stop stirring and allow the product to settle. Once the product has settled, decant the solution and place it in the waste bottle that can be found in the fume hood. Be careful not to lose any solid. Next wash the solid with hot deionized water. After the solid has settled, decant off the wash and place it in the waste bottle. Save the solid for the next step.

Question D: Write the balanced chemical equation for the reaction in this step. Assume the products are copper(II) oxide and water.

4. Formation of Copper(II) Sulfate:

Copper(II) oxide will react with sulfuric acid to produce copper(II) sulfate. Perform this reaction using 3 M sulfuric acid. As with all reactions, use enough sulfuric acid to get the job done, but do not generate excess waste. In preparation for the formation of the solid copper, quantitatively transfer your solution from your 250 mL Erlenmeyer flask to a 250 mL beaker. The term quantitatively transfer indicates that after you have poured the solution out of your 250mL Erlenmeyer flask you should wash any solid left in the flask with a small amount of deionized water (less than 5 mL), adding the rinse to the solution in the 250 mL beaker with your solution. You should repeat this wash and rinse 2 more times. The decantant can be poured down the sink with copious amount of water.

Now move back to the fume hood.

Question E: Write the balanced chemical equation for this reaction. Assume the products are copper(II) sulfate and water.

5. Formation of Copper Metal:

Working in the fume hood, quickly add 1.0 g of zinc metal and stir. The reaction will be complete when the solution is colorless. Record your observations. When the reaction is complete, decant the solution into a clean beaker. Examine the solid, if you note any solid zinc then add 10 mL of 6 M HCl and stir the solution until the

Reactions of Copper

DATA ANALYSIS

zinc is no longer present. When gas is no longer being produced, decant this solution. The decantant should be poured into the cation metal waste container.

Question F: Write the balanced chemical equation for the reaction in this step. Assume the reactants are copper(II) sulfate and zinc, and that the products are copper metal and zinc sulfate.

Question G: Indicate the reducing agent and the oxidizing agent in this reaction.

Question H: Was the reaction between zinc and hydrochloric acid endothermic or exothermic? What was the gas produced in the reaction?

6. Recovery of Copper Metal:

You will now transfer the copper from the beaker into the casserole dish. First weigh your dry casserole dish. Using your wash bottle, transfer all the solid to the casserole dish. Stir the solid in the water to wash away water soluble impurities. Decant the water and wash two more times with 5 mL aliquots of deionized water. Carefully and slowly heat the casserole on a hot plate. Watch this carefully to keep the solid from bumping. Continue the heating and stir the precipitate vigorously. Once the solid is completely dry, allow the casserole to cool, and weigh the casserole.

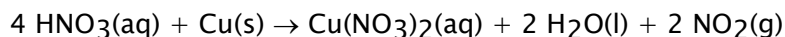
Question I: Calculate the percent yield you obtained. This can be found by dividing the actual yield you obtained by the theoretical yield. The theoretical yield is the ideal yield you would have if there were no loss or contamination during the experiment. The actual yield is the yield you actually obtained in the experiment.

Question J: Give reasons as to why you might expect your percent yield to be low. Give reasons as to why you might expect the percent yield to be high. Explain why your percent yield is high (>100%) or low (<100%)?

Clean-Up. Clean all glassware that was used before leaving the laboratory. Clean the casserole by soaking it in 6 M nitric acid solution in the hood. **Do not use concentrated nitric acid** for this step.

DATA ANALYSIS

In step 1 of the experimental procedure, copper metal is added to concentrated nitric acid. The reaction between copper metal and concentrated nitric acid is an oxidation-reduction reaction that is somewhat complicated.



Identify the element or elements that undergo OXIDATION. Identify the element or elements that undergo REDUCTION. Considering which elements are being oxidized and reduced, determine how many moles of electrons are transferred between the elements being oxidized and reduced.

In step 2 of the experimental procedure, you added 6 M sodium hydroxide to the copper(II) nitrate solution to form copper(II) hydroxide. You added a minimum amount of sodium hydroxide, but enough to complete the reaction. How do you determine that enough sodium hydroxide has been added?

In step 2 of the experimental procedure, you added 6 M sodium hydroxide to the copper(II) nitrate solution to form solid copper(II) hydroxide. What is the BALANCED chemical equation for the reaction in this step?

In step 3 of the experimental procedure, the copper(II) hydroxide was heated and decomposed to copper(II) oxide and water. What is the BALANCED chemical equation for this reaction?

In step 4 of the experimental procedure, 3 M sulfuric acid is added to the copper(II) oxide to produce copper(II) sulfate and water. What is the BALANCED chemical equation for this reaction?

In step 5 of the experimental procedure, 1.0 g of zinc is added to the copper(II) sulfate solution to produce copper metal and zinc sulfate. What is the BALANCED chemical equation for this reaction?

In step 5 of the experimental procedure, 1.0 g of zinc is added to the copper(II) sulfate solution to produce copper metal and zinc sulfate. What are the oxidizing and reducing agents?

In a 2nd reaction in step 5 of the experimental procedure, 6 M HCl is added to copper metal to remove any excess zinc metal. Is the reaction between the HCl and Zn exothermic or endothermic and what gas is released in this reaction? Hint: An exothermic reaction releases heat and an endothermic reaction absorbs heat.

In step 1 of the experimental procedure, you were instructed to weigh between 0.500g and 0.530g of copper wire. Enter the precise mass of your copper wire. Report your mass to a thousandth of a gram, i.e. 0.512 g.

In the final step of the experimental procedure, you dried your recovered copper. Enter your precise mass of the recovered copper. Report your mass to a thousandth of a gram, e.g. 0.512 g.

Calculate the percent yield you obtained. Report your percentage to 3 significant digits, e.g. 89.3%.

What factors would cause the percent yield to be LOW?

What factors would cause the percent yield to be HIGH?

Summarize the main results of this week's experiment. Explain why your percent yield is greater than 100% or lower than 100%.

General Analytical Techniques

INTRODUCTION

Analytical chemistry enables us to identify the constituents of unknown compounds and the relative amounts of these constituents. When we identify an unknown compound, we are performing a *qualitative analysis*. On the other hand if we can numerically determine the amounts of constituents in an unknown sample, we are performing a *quantitative analysis*. For example, suppose you were asked to determine what had caused a large fish kill in a lake or river. You would first attempt to determine **what** is in the water to see what could have caused the disaster. This is *qualitative analysis*. Then you would determine **how much** of the suspected toxin was present to determine if the concentration was high enough to cause the problem. This is *quantitative analysis*. In this lab you will learn some of the techniques used in *quantitative analysis*.

Quantitative analysis offers a numerical description of the amount of a constituent (the analyte) in a sample. In this lab we will express our measurements in molarity - *moles of analyte per liter of solution*. For your information you should be aware that often a more precise measurement is needed. When testing for toxicities, a thousandth of a gram can make the difference between a safe amount and one that is lethal. These measurements are often taken in parts per million, that is, one microliter of analyte per liter of sample!

Over the next two weeks, you will have the opportunity to use two common analytical laboratory techniques. One of the procedures you will perform involves *volumetric analysis* using titration methods. You will need to prepare solutions of a specific concentration using volumetric glassware and graduated burets. In a volumetric analysis, you measure the volume of solution that contains a sufficient amount of reagent to react completely with the analyte.

Another of the procedures you will perform involves *spectroscopic analysis*, using a GENESYS™ spectrophotometer. Using this method, you will need to focus on the proper preparation of aqueous solutions from solids and water, and on the process of diluting a concentrated solution.

Some of these methods are more precise than others, but they will all be imprecise if your technique is poor. Try to be as accurate and as careful as possible. Also, be careful when using volumetric glassware as it is extremely expensive; handle all volumetric glassware with great care to avoid breakage. Before beginning the analytical experiments you should first become acquainted with common laboratory procedures. You must do this by reading the "Introduction" section of this manual. In addition, many of the calculations must be done before entering the laboratory.

In doing these experiments you should keep in mind that the goal is to learn to make solutions properly, and to become acquainted with a couple of common analytical techniques. It is important to learn these procedures properly now so as to avoid costly errors later. You should also begin to understand that certain analytical techniques are more accurate than others.

Volumetric Analysis

INTRODUCTION

In Part I of this experiment you will prepare a solution of sodium hydroxide and *standardize* it against very pure potassium acid phthalate in a series of replicate titrations. Potassium acid phthalate (KHP) is a monoprotic acid with the formula $\text{KHC}_8\text{H}_4\text{O}_4$. You will then use the standardized sodium hydroxide solution to determine the acetic acid content of a dilute unknown solution.

A standard solution is one whose solute concentration is accurately known. If a solute can be obtained in a very pure, stable, weighable form, a *primary standard* solution of it can be prepared directly. This is done by dissolving an accurately determined amount of the solute in the desired solvent and bringing the total volume of the solution to an accurately known final value. Care must be taken to insure that the solution is homogeneous and that it is at ambient temperature when the final adjustment of its volume is made.

If the desired reagent cannot be obtained in primary standard form, one can only prepare a secondary standard solution of it. A solution of the reagent having approximately the desired concentration is prepared which is then standardized (1) by titration against a measured mass of a suitable primary standard substance; or (2) by titration against another reliably known secondary standard solution; or (3) by direct analysis for the reagent in question by some suitable non-titrimetric method such as spectroscopic analysis.

We shall use standardization method (1) in Part I of this experiment utilizing potassium hydrogen phthalate as the primary standard to prepare a secondary standard solution of sodium hydroxide.

In Part II of this experiment you will prepare a solution of hydrochloric acid and *standardize* it against the sodium hydroxide solution you prepared in Part I. The standardized hydrochloric acid solution is referred to as a tertiary standard. You will use it to determine the neutralization capacity of commercial Antacid tablets.

Antacids work by neutralizing excess acid in your stomach. The active ingredient is usually carbonate ion (CO_3^{2-}) or hydroxide ion (OH^-). In this experiment, you will determine the mass and percentage of hydroxide or "equivalent hydroxide" that is present in the tablet, where one mole of carbonate is equivalent to the neutralizing capacity of two moles of hydroxide. You will accomplish this by first dissolving the antacid sample in excess HCl (because many antacids do not dissolve in water alone). Some, but not all, of the added HCl will react with the base present in the tablet. You will then titrate the solution with NaOH to determine how much HCl is remaining or is in excess. By difference you will then be able to calculate the amount of HCl that reacted with the antacid and thus the neutralization capacity of the antacid tablet. Using the calculated amount of HCl that reacted, you may also determine the mass and percentage of hydroxide or "equivalent hydroxide" in the tablet. One lesson you will learn from this part of the

Volumetric Analysis

PART I. PROCEDURE

experiment is that the analyses of "real life" unknowns are often complex and more difficult.

Safety. Wear goggles throughout the entire experiment, especially when reading a buret! Be especially careful with acids and bases. All waste from this experiment can be poured down the drain with copious amounts of water.

Work in pairs on this 2-day experiment.

Stock Chemicals Used

Chemical	Maximum Amount Used
50% (w/w) Sodium Hydroxide	According to calculation
6M Hydrochloric Acid	According to calculation
Potassium Hydrogen Phthalate	3 g
1% Phenolphthalein Indicator	Drops
Methyl Orange Xylene Cyanol FF	Drops
Acetic Acid Unknowns A, B, C, D	~ 20 mL

PART I. PROCEDURE

Analysis of an Unknown Acetic Acid Solution

A. Preparing the Sodium Hydroxide Solution

In this step of the experiment you will prepare 500 mL of approximately 0.15 M sodium hydroxide solution by diluting a commercial stock solution that is 50% NaOH by mass and has a density of 1.525 g/mL (grams of solution per mL of solution).

1. Begin by pouring about 200 mL of deionized water into your (clean) plastic bottle. Calculate the appropriate volume of stock solution, use a polyethylene dropper to dispense it into a 25 mL graduated cylinder, and then pour the contents of the graduate into the partially filled plastic bottle. Rinse the graduate out with fresh water at least twice, adding the rinsings to the contents of the plastic bottle. Screw the cap on the plastic bottle and mix the contents thoroughly by inverting the bottle and swirling it repeatedly. Then add the remaining 300 mL of water in three 100-mL batches, mixing the bottle contents thoroughly each time. The bottle should be shaken at least 30 times after the last addition.

B. Standardizing the Base Against Potassium Acid Phthalate

In this step of the experiment you will standardize your sodium hydroxide solution against the primary standard, potassium acid phthalate. You will also use a technique called *weighing by difference*. This is a very important technique to use because it eliminates systematic errors from the balance. Weighing by difference is quite simple. First, mass the container and the material from which you are going to draw your sample. Then remove some of the material and place it in a separate container. Remeasure the mass of

the original container and the remaining material. Calculate the mass removed, and repeat the process until you have removed the mass desired.

Question A: Explain how weighing by difference eliminates systematic balance errors.

2. In a previous laboratory period you will have obtained about 3.0 grams of primary-standard grade potassium acid phthalate (KHP) in a vial, dried it in an oven at 110°C for 2 hours, and stored it in a desiccator for use today.
3. In what follows, use a folded paper strip to handle the vial; this will keep oils from your hands from changing the mass of the vial. Accurately weigh a 0.5-0.6 gram sample of dry KHP on to a weighing boat. Using the appearance of this sample as a guide, accurately weigh two or three more such samples by difference into clean 125 or 250 mL Erlenmeyer flasks. Quantitatively transfer the first sample from the weighing boats into a 500 mL Erlenmeyer flask with the help of a small stream of water from your wash bottle, and then add about 35 mL of water to each of the three flasks and swirl them gently until the solids dissolve. Be careful not to leave drops of the solution on the side walls of the flask.
4. With the technique described in the Common Laboratory Procedures section of this manual, rinse and fill a 25 mL buret with the base solution you wish to standardize. Do not waste time trying to hit 0.00 with the meniscus. Rather, go a few drops below the zero mark and read and record the actual starting point to the nearest 0.02 mL. 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.
5. Add three drops of phenolphthalein indicator to the first KHP solution. Place the flask under the buret and lower the buret tip well into it. The first titration should be a cursory titration to find the approximate volume of the end point. Place a piece of white notebook paper under the flask and add about 2 mL at a time until the solution turns a permanent pink. The solution should be stirred continuously. Note the volume needed to reach the end point. Refill your buret and record the initial buret reading. Now, perform the second titration quickly but more carefully. Open the stopcock somewhat and allow a gentle but steady stream of base to flow (without splashing) into the pooled acid solution. You can add the base fairly rapidly at first, but pay attention to the region where the two solutions mix in the flask, and as the pink color which forms there begins to tail out into the solution as you stir the contents of the flask. Stop rapidly adding the base when you are within 2 mL of the approximate endpoint. Gently wash down the walls of the flask with water from your wash bottle, and then resume adding base from the buret but now dropwise. As you approach the endpoint, the pink color will increasingly linger. You should frequently wash down the interior sides of the flask to recover any reagent drops from clinging to the sides. Swirl the flask after the addition of each drop or ½ drop. A ½ drop may be added by slowly opening the stopcock until a ½ drop forms at the tip. Wash this ½ drop into the flask. Stop adding base when the entire flask has a *faint* pink color that persists. You may wish to record the buret

Volumetric Analysis

volume of several successive drops as you approach the endpoint in case you discover that you have overshoot the endpoint. Record the final buret reading to the nearest 0.02 mL. Refill the buret and similarly titrate the remaining KHP samples.

6. It should be clear to you that the ratio of the NaOH titration volume to the mass of KHP being titrated should be a constant. Calculate this ratio for your three titrations and determine if one of them fails the Q-test. If it does, run another sample. When you have three samples that can be retained, calculate and report the average molarity of the NaOH solution, the 90% confidence interval for the reported molarity, and the relative average deviation of the your measurements in parts per thousand. NOTE: KHP has the actual chemical formula $\text{KHC}_8\text{H}_4\text{O}_4$, formula mass = 204.23.

Question B: Why doesn't it matter how much water you add when dissolving the acid or when carrying out the titration?

C. Analysis of the Unknown Acetic Acid Solution

7. Obtain approximately 20 mL of an acetic acid unknown in a clean 50-mL Erlenmeyer flask. Record the unknown number in your laboratory notebook.
8. Pipet 5.00 mL samples of acetic acid unknown into each of three clean 125, 250 or 500 mL Erlenmeyer flasks and wash down the sides of the flasks with another 25 mL or so of water from your wash bottle.
9. One after the other, add three drops of phenolphthalein indicator to each of the titration vessels and titrate their contents with your standardized NaOH solution. A titration thief would probably be advisable for at least the first titration. Ideally, the range of the replicate titration volumes should be only one or two drops. One drop from these burets is about 0.05 mL, so two "5 mL" titration volumes that differ by 2 drops disagree with each other by 1%.
10. Calculate and report the average titratable acid concentration, $[\text{HC}_2\text{H}_3\text{O}_2]$, in the original unknown solution and the 90% confidence interval for this result. Also calculate and report your average value for the mass percentage of acetic acid in the original unknown solution along with the 90% confidence interval for this result. Assume a density of 1.00 g/mL for the acetic acid unknown.

Clean-up. After Part I. of the experiment is completed, drain any remaining solution from the buret. Rinse each buret with deionized water. Then, leaving the stopcock open, return the inverted burets to the buret clamp.

SAVE YOUR REMAINING NaOH FOR PART II OF THIS EXPERIMENT!

PART I. DATA ANALYSIS

How many grams of pure, solid NaOH are required to make 500 mL of 0.15M NaOH solution? .

How many grams of a solution that is 50% by weight NaOH is required to make 500 mL of 0.15M NaOH solution? g.

The density of a 50% solution of NaOH is 1.525 g/mL. What volume of a solution that is 50% by weight NaOH is required to make 500 mL of 0.15M NaOH solution?

Why does weighing by difference eliminate systematic balance errors?

For each of your 3 acceptable trials (not the cursory one) in standardizing your NaOH against KHP, what are the mass in grams of KHP for each of the three samples to one thousandth of a gram, e.g. 0.507g?

What is the volume in mL of NaOH required to reach the endpoint for each of the KHP samples and enter the volume to a hundredth of a mL, e.g. 15.24 mL?

Calculate the values for each of your three trials for the molarity of your NaOH solution. Place your molarity entries in the order corresponding with the masses of KHP and the volumes of NaOH required for the trials.

Calculate the value for the average of the three trials determining the molarity of your NaOH solution.

Calculate the value for the standard deviation of the average molarity.

Calculate the value for the 90% confidence limits for the molarity of your NaOH solution.

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

What was the letter of your unknown sample of acetic acid?

What volume in mL did you use for each of the three acceptable trial samples of your unknown solution of acetic acid?

What was the volume of standardized NaOH solution that was required for each trial determining the amount of acetic acid in your unknown?

Calculate the molarity of your three acetic acid trials using the volume of standardized NaOH solution required for each and the average molarity of the NaOH solution from the standardization trials with KHP.

Calculate the value for the average of the three trials determining the molarity of your unknown acetic acid solution.

Calculate the value for the standard deviation of the three trials for the molarity of your unknown acetic acid solution.

Volumetric Analysis
PART I. DATA ANALYSIS

Calculate the value for the 90% confidence limits for the molarity of your unknown acetic acid solution.

PART II. PROCEDURES**Neutralization Capacity of Commercial Antacid Tablets****A. Preparing the Hydrochloric Acid Solution**

In this procedure the secondary standard NaOH solution will be used to standardize an HCl solution to form a tertiary standard.

1. You will prepare 200 mL of ~0.6 M HCl from a 6.0 M stock solution in a 500 mL Erlenmeyer flask. Calculate how much 6.0 M HCl you will need and roughly how much deionized water will be required. Place about 80% of the required deionized water into the 500 mL Erlenmeyer flask. Measure the calculated amount of 6.0 M HCl using a graduated cylinder and transfer this volume to the flask. Now add deionized water to the 200 mL mark on the Erlenmeyer flask. Cover the flask with Parafilm and mix well. You have now successfully prepared the acid solution. Next, you will determine the true concentration of your acid.

Question C: You always add acid to water, and never the reverse. Why?

B. Standardizing the Acid Against Sodium Hydroxide

2. Accurately transfer 5.00 mL of your prepared hydrochloric acid solution using a clean volumetric pipet into a clean Erlenmeyer flask and add approximately 20 mL of deionized water to the flask to allow you to swirl the contents easily. Rinse and fill a 25 mL buret with your standard NaOH solution. Make three or four samples. Add 2-3 drops of phenolphthalein indicator to the flask and titrate to the endpoint as described in step 9 of Part I. Record the final volume of base. Repeat this titration at least two more times. You will need to reuse one of your Erlenmeyer flasks. Calculate the actual concentration of your hydrochloric acid solution using the concentration of base you determined in Part I for each trial.
3. Calculate the average concentration of your acid and the relative and standard deviation of your data.

C. Antacid Analysis

4. Prepare 2 burets for your antacid titration. Condition and fill a 25 mL buret with standard acid. Make sure the buret is thoroughly washed since it may have previously contained base. Condition and fill a 25 mL buret with your standard base. You may label the buret holder in order to be sure to remember which buret contains acid and which contains base. Record the initial buret readings.
5. Obtain antacid tablets from one of the brands provided in the laboratory. Record the masses of the tablets in your notebook. Crush the first antacid tablet using a mortar and pestle. Suggested masses to be used for analysis are indicated on the antacid containers. Record the mass of the crushed sample in your laboratory notebook. Quantitatively transfer the sample to a clean 125 mL Erlenmeyer flask.

Volumetric Analysis
PART II. DATA ANALYSIS

6. Add approximately 25 mL of deionized water to the flask. Carefully and accurately dispense 12.5 mL of your standard acid into the 125 mL flask using your buret. Next clamp the flask and heat the contents with a hot plate. Boil the solution for 5 minutes. The sample may not completely dissolve during this process due to the presence of "fillers", but any base will have reacted with the excess HCl. Using your alkacid paper, check to be sure that all base has reacted, and that there is excess acid present. If the solution is not acidic, then accurately add another 12.5 mL of acid and boil for another 5 minutes.

Question D: Write a balanced chemical equation that describes the reaction if the antacid contains hydroxide ion.

Question E: Write a balanced chemical equation that describes the reaction if the antacid contains carbonate ion.

7. Cool the solution to room temperature by carefully immersing the flask in a container of tap water. Add 5 drops of methyl orange/xylene cyanole FF indicator (MOXC) to the solution. Titrate the sample with your NaOH standard. The indicator MOXC goes from red (acid), through grey, to green (base). The grey color is the end-point. The end point is not as obvious as that of phenolphthalein. If you add too much base, you will need to back-titrate with the standard HCl. That is, you will have to add a carefully measured volume of standard HCl to return to the red color. You can then titrate again to the grey end-point using the standard base.
8. Repeat the experiment using the leftover mass from the brand you selected. Report the neutralization capacity (millimoles of HCl neutralized) of the antacid tablet you used. Calculate the mass and percentage of hydroxide or "equivalent hydroxide" per gram of antacid tablet.
9. Calculate an average, relative deviation, and standard deviation. Report the results in your notebook. Be sure to show all calculations.

Clean-up. Drain any remaining solution from the burets. All waste from this experiment can be poured down the drain. Rinse each buret with deionized water and return to the buret shelf.

PART II. DATA ANALYSIS

What volume of 6 M HCl is needed to prepare 200 mL of 0.6 M HCl?

What volume in mL of your HCl solution did you use for each of the three acceptable trial samples for the standardization? Your volume of HCl should be approximately 5 mL.

What was the volume of standardized NaOH solution that was required for each trial for the standardization of HCl?

Calculate and report the molarity of your three HCl standardization trials using the volume of standardized NaOH solution required for each and the average molarity of the NaOH solution from the standardization trials with KHP.

Report the value you calculated for the average of the three trials determining the molarity of your HCl solution.

Report the value you calculated for the standard deviation of the three trials for the molarity of your HCl solution.

Report the value you calculated for the relative deviation, in parts per thousand, of your HCl solution.

Report your measured masses of the antacid tablets.

For each of the three trials you performed for the analysis of an antacid tablet, what was the mass of the crushed portion of the tablet that you dissolved in your standardized HCl?

For each of the three trials you performed for the analysis of an antacid tablet, what volume (in mL) of tertiary standard HCl did you use to dissolve the samples?

Report the total volume of acid that you used to dissolve the samples, including any additions of acid after the first one needed to make the solution acidic, to the nearest 0.02 mL.

For each of the three trials of the analysis of the antacid, what was the volume of standardized NaOH required to titrate the residual standardized HCl after it reacted with the antacid?

Based upon the molarities of the standardized HCl and standardized NaOH, and the volumes of each added to analyze the antacid samples, calculate the MILLIMOLES of HCl per MILLIGRAM of sample.

Using the equivalent millimoles HCl/mg sample and the mass of the tablet, calculate the equivalent millimoles HCl neutralized per tablet for each of your three trials.

Using the equivalent millimoles HCl/mg sample, the stoichiometry of reaction of HCl with NaOH, and the molar mass of the hydroxide ion, report the gram equivalent OH^- per gram of antacid for each of your three trials.

Convert the gram equivalent OH^- /gram antacid to % equivalent OH^- for each of your trials.

Report the value you calculated for the average of the three trials for % equivalent OH^- .

Report the value you calculated for the standard deviation of the three trials for the % equivalent OH^- .

Volumetric Analysis
PART II. DATA ANALYSIS

Report the value you calculated for the relative deviation, in parts per thousand, of your % equivalent OH⁻.

Write a summary of this experiment in a paragraph or two describing the goal, procedures undertaken, results, and sources of error in the experiment.

Spectrophotometry (Part I & II)

INTRODUCTION

In this experiment you will be performing a spectroscopic analysis of known and unknown compounds and mixtures. You will be using the GENESYS™ 20 to find the concentration of an unknown mixture of Bromothymol Blue and Thymol Blue.

A Brief Introduction to Spectrophotometry

The use of light to measure chemical concentrations is called spectrophotometry. When you pass light through a sample, a certain amount of light is absorbed by the molecules in solution, and the rest passes through. In fact, materials have specific color due to certain wavelengths or colors of the visible spectrum that are being absorbed by the molecules in the material. The color you attribute to the material is actually all the remaining reflected light. If we measure the amount of light that is going into the sample and we measure the amount of light that comes out, we can determine to what extent the sample is absorbing the light.

In this experiment you will carefully analyze the concentration of solutions of Bromothymol Blue and Thymol Blue. To do this you will use a spectrophotometer, an instrument which measures the fraction of light (I/I_0) that is absorbed by a sample. The sample is placed in a tube called a cuvette and is then irradiated with an incident beam of light (I_0) of a specific wavelength. A detector then measures the amount of light that is transmitted through the sample (I). A schematic representation of a spectrophotometer is shown below in Figure 1.

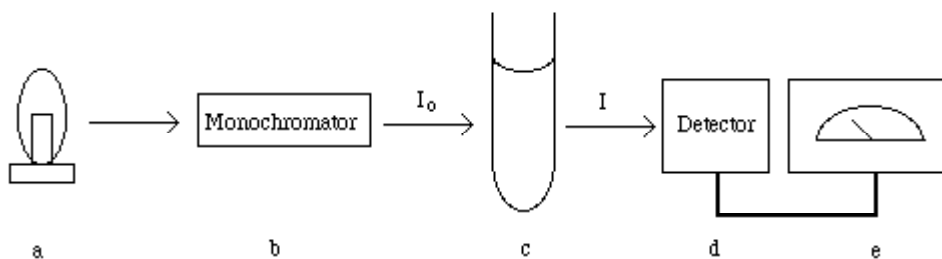


Figure 1. A schematic representation of a spectrophotometer.

- (a) A light source produces a polychromatic beam of light. The source used for this experiment produces light of wavelength ranging from 340–1000 nm.
- (b) The monochromator selects a particular wavelength for the incident beam of light (I_0).
- (c) This is the sample cell. Treat the Cuvettes with care.
- (d) The detector measures the intensity of the transmitted light (I).
- (e) The readout is supplied by a meter.

Spectrophotometry (Part I & II)

INTRODUCTION

For any substance, the amount of light absorbed depends on: 1) the concentration of the absorbing species, 2) the length of solution that the light passes through (commonly called the path length), 3) the wavelength of incident light, and 4) the identity of the absorbing species present.

One of the more common ways of expressing the amount of light absorbed is called percent transmittance, %T, which is defined for a particular wavelength as

$$\%T = (I/I_0) \times 100$$

Since the solvent itself may absorb light of the wavelength selected, the value of I_0 will be chosen as the intensity of light transmitted when the cuvette is filled with solvent only. Another common method of expressing the amount of light absorbed is called absorbance. Absorbance is defined by

$$A = \log_{10} (I_0/I) = \log_{10} (100 / \%T)$$

Consider a completely transparent sample. In this case the percent transmittance would be 100 which corresponds to an absorbance of 0. Also note that a percent transmittance of 50 corresponds to an absorbance of 0.301. Although absorbance may at first appear complex, it is a very useful measurement. This is because it is directly related to concentration as is shown in the following equation, called Beer's Law:

$$A = \epsilon \ell C$$

where A is the absorbance, c is the molar concentration, ℓ is the path length, and ϵ is the molar absorptivity or molar extinction coefficient. Note that a plot of A vs. c will yield a straight line which has a slope of $\epsilon \ell$. Be aware that Beer's Law does not necessarily hold for all substances or at all concentrations; for example, it often fails at high concentrations.

In this lab you will be finding the concentration of an unknown solution of Bromothymol Blue and Thymol Blue. Knowing that $\epsilon \ell$ is the slope of the line for the graph of A vs. c , then you should be able to find the concentration experimentally. That is, if you find the absorbance of Bromothymol Blue and Thymol Blue at different concentrations and plot this set of data, you should get a linear plot that has a slope of $\epsilon \ell$.

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.

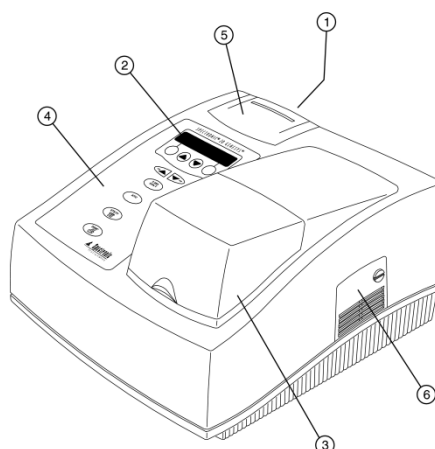
The calibration procedure entails setting 0 Absorbance at a given wavelength with a cuvette containing a reference or blank solution. The blank solution is missing the component of interest, but is otherwise as identical as possible to the solution to be analyzed for the component of interest. Typically,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

Spectrophotometry (Part I & II)

INTRODUCTION

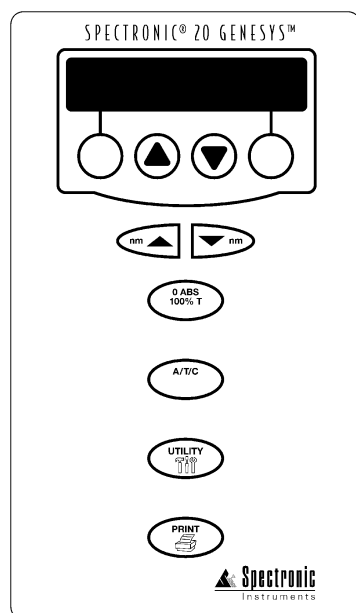
GENESYS™ 20 Spectrophotometer

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.

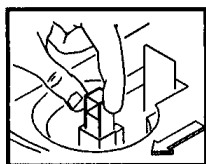


1. Press **A/T/C** to select the absorbance or % transmittance mode. The current mode appears on the display.



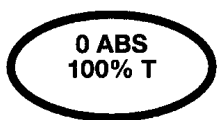
2. Press **nm ▲** or **nm ▼** to select the wavelength.

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

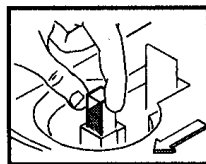


3. Insert your blank into the cell holder and close the sample door.

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.

Spectrophotometry (Part I & II)

INTRODUCTION

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 the 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 pH 8.0 phosphate buffer 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 dials 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.

Spectrophotometry (Part I & II)
PART I PROCEDURE

Safety: Treat the GENESYS™ 20 spectrophotometers with great care as they are expensive and delicate instruments. Wear your goggles.

PART I PROCEDURE

You will work in pairs on this experiment.

Stock Chemicals Used

Chemical	Maximum Amount Used
Bromothymol Blue Standard	80 mL
Thymol Blue Standard	80 mL
Conc. pH 8.0 Buffer	50 mL
1 st Week: Bromothymol Blue Unknown	4.5 mL
2 nd Week: Mixture Unknown	4.5 mL

Solution Preparations

Preparation of the Bromothymol Blue Solutions

1. Make a diluted stock solution of the pH 8.0 buffer by measuring 50 mL of pH 8.0 buffer into a clean Erlenmeyer flask and dilute with water to the 250 mL graduated mark. (The 5% accuracy of the glassware used is more than adequate. Other solutions will need much greater accuracy.) This reagent will be used as a solvent in preparation of all additional reagents

Two Important Notes:

Make all your dilutions in steps 3 – 6 **using only** the two 25 mL volumetric flasks in your locker. Plan your work accordingly.

Do not use the stock “Buffers Solutions” at pH 4, 7, and 10 in the boxes at the side

2. Bromothymol Blue (molecular weight 646.38) – 23.2 μ M stock solution in a pH 8.0 phosphate buffer will be used to prepare the solutions. (*This is standard solution #1*)
3. Use a 5 mL volumetric pipet to deliver 5.00 mL of diluted pH 8.0 buffer into a 25 mL volumetric flask. Fill to the mark with 23.2 μ M Bromothymol Blue. (*This is standard solution #2*)
4. Use a 5 mL and a 10 mL volumetric pipet to deliver 15.00 mL of the 23.2 μ M Bromothymol Blue into a 25 mL volumetric flask. Fill to the mark with the pH 8.0 buffer. (*This is standard solution #3*)
5. Use a 10 mL volumetric pipet to deliver 10.00 mL of the 23.2 μ M Bromothymol Blue into a 25 mL volumetric flask. Fill to the mark with the pH 8.0 buffer. (*This is standard solution #4*)

6. Use a 5 mL volumetric pipet to deliver 5.00 mL of the 23.2 μM Bromothymol Blue into a 25 mL volumetric flask. Fill to the mark with the pH 8.0 buffer. (*This is standard solution #5*)

Preparation of the Thymol Blue Solutions

7. You will use Thymol Blue (molecular weight 488.58) – 61.6 μM stock solution in a pH 8.0 phosphate buffer.
8. Using the stock solution of Thymol Blue, prepare a series Thymol Blue standard solutions using the same method outlined for Bromothymol Blue in steps 3 – 6.
9. You have now made a total of 10 samples. Calculate the concentration of each and record the data in your lab notebook. Your TA will check off your concentrations before you leave.

Spectral Scans of Bromothymol Blue and Thymol Blue

10. Calibrate the GENESYS™ 20 spectrophotometer as described in the discussion above.
11. Set the wavelength to 370 nm.
12. Fill a cuvette with pH 8.0 phosphate buffer as the blank.
13. Fill a second cuvette with 23.2 μM Bromothymol Blue and measure the absorbance
14. Fill a third cuvette with 61.6 μM Thymol Blue and measure the absorbance.
15. Use the same three samples to measure the absorbance at increasing wavelengths. For each measurement increase the wavelength increment by 10 nm from 370 nm to 670 nm (i.e., 370 nm, 380 nm, 390 nm, ... , 670 nm). Also, measure the absorbance at 434 and 618 nm during this data collection.
16. Make a rough plot during the laboratory period of Bromothymol Blue vs. wavelength and Thymol Blue vs. wavelength. For each graph, add the absorbance values at each particular wavelength to create a new graph of absorbance (sum of Bromothymol Blue and Thymol Blue) vs. wavelength. After the laboratory, make a clear plot using Excel.

Question: What does each graph represent? The answer to this question will prepare you for Part II of this laboratory next week. Include an answer of this question in your Post-laboratory conclusion statement.

Spectrophotometry (Part I & II)

PART I DATA ANALYSIS

Measuring Absorbances of the Bromothymol Blue Solutions

17. Calibrate the GENESYS™ 20 spectrophotometer as described in the discussion above. Remember to use the pH 8.0 phosphate buffer for the blank.
18. Using the reference standards (steps 2 – 6) for Bromothymol Blue, measure the absorbances at 618 nm (the absorption maximum of Bromothymol Blue). Record all five absorbance readings in your notebook.
19. Obtain a Bromothymol Blue solution with unknown concentration from the TA. Record the unknown letter in your notebook. Measure the absorbance at 618 nm using the GENESYS™ 20 and record the data.

Making your Beer's Law Plot for Bromothymol Blue

20. Make a Beer's law plot of Absorbance vs. Concentration using the data from the data collected in step 18. Make a rough plot during the laboratory period to see if the graph is linear. If not, re-measure a point that does not fit the line. After the laboratory, make a clear plot using Excel. Include the best-fit line for your data that goes through the origin. Find the slope of your graph. Using 1.0 cm for l , calculate the value of ϵ in units of $\mu\text{M}^{-1}\text{cm}^{-1}$.
21. Determine the concentration of the unknown solution of Bromothymol Blue by using the Beer's law plot and the measured concentration.

SAVE YOUR 10 SAMPLES FOR PART II

PART I DATA ANALYSIS

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

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

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

Report the precise concentration of the undiluted stock solution #1 of the Bromothymol blue in micromoles per liter. This is your most concentrated (undiluted) standard solution for which you measured the absorbance.

Report the precise volume of the Bromothymol blue standard solution #1 in milliliters used to prepare standard solution #2.

You diluted a portion of the Bromothymol blue standard solution #1 with the pH 8.0 buffer to make standard solution #2. Report the total volume of the resulting Bromothymol blue standard solution #2 you prepared.

Calculate the molarity of Bromothymol blue standard solution #2.

Report the precise volume of the Bromothymol blue standard solution #1 in milliliters used to prepare standard solution #3.

You diluted a portion of the Bromothymol blue standard solution #1 with the pH 8.0 buffer to make standard solution #3. Report the total volume of the resulting Bromothymol blue standard solution #3 you prepared.

Calculate the molarity of Bromothymol blue standard solution #3.

Report the precise volume of the Bromothymol blue standard solution #1 in milliliters used to prepare standard solution #4.

You diluted a portion of the Bromothymol blue standard solution #1 with the pH 8.0 buffer to make standard solution #4. Report the total volume of the resulting Bromothymol blue standard solution #4 you prepared.

Calculate the molarity of Bromothymol blue standard solution #4.

Report the precise volume of the Bromothymol blue standard solution #1 in milliliters used to prepare standard solution #5.

You diluted a portion of the Bromothymol blue standard solution #1 with the pH 8.0 buffer to make standard solution #5. Report the total volume of the resulting Bromothymol blue standard solution #5 you prepared.

Calculate the molarity of Bromothymol blue standard solution #5.

Report the precise concentration of undiluted stock standard solution #1 for Thymol blue in micromoles per liter. This is your most concentrated undiluted standard solution for which you measured the absorbance.

Report the precise volume of the Thymol blue standard solution #1 in milliliters used to prepare standard solution #2.

You diluted a portion of the Thymol blue standard solution #1 with the pH 8.0 buffer to make standard solution #2. Report the total volume of the resulting Thymol blue standard solution #2 you prepared.

Calculate the molarity of Thymol blue standard solution #2.

Report the precise volume of the Thymol blue standard solution #1 in milliliters used to prepare standard solution #3.

You diluted a portion of the Thymol blue standard solution #1 with the pH 8.0 buffer to make standard solution #3. Report the total volume of the resulting Thymol blue standard solution #3 you prepared.

Calculate the molarity of Thymol blue standard solution #3.

Report the precise volume of the Thymol blue standard solution #1 in milliliters used to prepare standard solution #4.

Spectrophotometry (Part I & II)

PART I DATA ANALYSIS

You diluted a portion of the Thymol blue standard solution #1 with the pH 8.0 buffer to make standard solution #4. Report the total volume of the resulting Thymol blue standard solution #4 you prepared.

Calculate and enter the molarity of Thymol blue standard solution #4.

Report the precise volume of the Thymol blue standard solution #1 in milliliters used to prepare standard solution #5.

You diluted a portion of the Thymol blue standard solution #1 with the pH 8.0 buffer to make standard solution #5. Report the total volume of the resulting Thymol blue standard solution #5 you prepared.

Calculate the molarity of Thymol blue standard solution #5.

Report your absorbance readings for the five Bromothymol blue standard solutions, beginning with the most concentrated solution.

Plot absorbance versus concentration in $\mu\text{mol/L}$ of the Bromothymol blue at the wavelength of its maximum absorbance on a spreadsheet or a programmable calculator, and carefully determine the slope of the best straight line fit through the data.

Report the letter / number of the sample of unknown concentration that was assigned to you by your TA.

Report the absorbance reading you measured for the Bromothymol blue solution of unknown concentration at the wavelength of its maximum absorbance.

Report the value you calculated for the concentration of your unknown sample of Bromothymol blue.

Determine the percent error in your analysis of the Bromothymol blue solution of unknown concentration.

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.

PART II. PROCEDURES

Spectral Assignments

Using Beer's Law Plots to Determine the Concentrations of Unknown Mixtures

1. Calibrate the GENESYS™ 20 spectrophotometer as described in the discussion above. Remember to use the pH 8.0 phosphate buffer for the blank.
2. Using the five reference standards for Thymol Blue, and measure the absorbances at 618 nm (the absorption maximum of Bromothymol Blue). Record the five measured absorbances in your notebook. The five Bromothymol Blue values were already measured at 618 nm in Part I.
3. Repeat step 2 for Thymol Blue and Bromothymol Blue at 434 nm (the absorption maximum of Thymol Blue).
4. When finished, you should have collected a total of **20 readings**.
5. The TA will provide an unknown. The unknown is a mixture of Bromothymol Blue and Thymol Blue. Subsequent data analysis will be used to determine the concentration of each substance in the mixture.

Scan the Unknown Mixture

6. Starting at 370 nm, measure and record the absorbance of the unknown mixture.
7. Increase the wavelength by 10 nm and measure and record the absorbance again. Remember to blank at each change in wavelength.
8. Repeat steps 6 and 7 until reaching 670 nm. In addition, measure the absorbance at 618 nm and 434 nm (Bromothymol Blue and Thymol Blue absorption maximum, respectively). Record all 33 measured values.
9. Plot Absorbance vs. Wavelength of the unknown mixture. Two peaks will be observed. Make a rough plot during the laboratory period. After the laboratory, make a clear plot using Excel.

Question: What do the two peaks in the plot of Absorbance vs. Wavelength represent? Include an answer of this question in your Post-laboratory conclusion statement.

Spectrophotometry (Part I & II)

PART II DATA ANALYSIS

PART II DATA ANALYSIS

Analysis using Beer's Law Plots

The data collected in Part II can be used to obtain the values of *effective molar absorptivity*, symbolized by ϵ^* , for both the Bromothymol Blue and Thymol Blue at the two wavelengths of interest here. Using that information you will calculate the concentration of each in the unknown.

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 $A = \epsilon^*C$. (Note that the way it is defined here ϵ^* must have dimensions M^{-1} because absorbance is dimensionless.)

We will distinguish between Bromothymol Blue and Thymol Blue using the following symbols in the equations:

BTB = Bromothymol Blue

TB = Thymol Blue

We shall henceforth distinguish the four different ϵ^* values by the subscripts listed below:

BTB,618 = Bromothymol Blue solution examined at the wavelength of the Bromothymol Blue absorption maximum (618 nm)

BTB,434 = Bromothymol Blue solution examined at the wavelength of the Thymol Blue absorption maximum (434 nm)

TB,618 = Thymol Blue solution examined at the wavelength of the Bromothymol Blue absorption maximum (618 nm)

TB,434 = Thymol Blue solution examined at the wavelength of the Thymol Blue absorption maximum (434 nm)

Determining Effective Molar Absorptivity

Calculate the molar concentrations of the Bromothymol Blue and Thymol Blue solutions you studied in Part I.

Prepare four graphs of absorbance vs. concentration. You must plot the data on an Excel spreadsheet. From these plots determine the four pertinent values of ϵ^* (slope of the graph).

Spectrophotometric Analysis of an Unknown Mixture of the Two Complexes

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 Bromothymol Blue and Thymol Blue compounds in significant amounts the absorbance at 618 nm (λ_{max} of Bromothymol Blue) will be given by the equation

$$A_{618} = \epsilon^*_{\text{BTB},618} [\text{C}_{\text{BTB}}] + \epsilon^*_{\text{TB},618} [\text{C}_{\text{TB}}]$$

and the absorbance at 434 nm (λ_{max} of Thymol Blue) will be given by the equation

$$A_{434} = \epsilon^*_{\text{BTB},434} [\text{C}_{\text{BTB}}] + \epsilon^*_{\text{TB},434} [\text{C}_{\text{TB}}]$$

where $[\text{C}_X]$ is the molar concentration of $X = \text{TB}, \text{BTB}$.

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 Bromothymol Blue and Thymol Blue in the mixture.

Scan Adjustments

Using the spectral scans of Bromothymol Blue and Thymol Blue from Part I (steps 15 & 16), adjust the measured absorbances to compare to the scan of the unknown mixture. An adjustment can easily be using the equation: $A_2 = (c_2/c_1) A_1$, where c_2 is the concentration of either Bromothymol Blue or Thymol Blue in the unknown mixture, c_1 is the concentration of either Bromothymol Blue or Thymol Blue from the spectral scans of Part I, and A_1 is the absorbance of either Bromothymol Blue or Thymol Blue from the spectral scans of Part I.

After making an adjustment to the spectral scans of Bromothymol Blue and Thymol Blue from Part I, then add the absorbance values at each particular wavelength to create a new graph. The graph is a plot of absorbance (sum of Bromothymol Blue and Thymol Blue) vs. wavelength. Plot this graph together with the graph created on step 8 of Part II. Discuss any questions about these graphs with your TA before leaving lab or at office hours.

Post-laboratory Questions

Report the 5 absorbance readings 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).

Plot absorbance versus concentration in $\mu\text{mol/L}$ of the Bromothymol blue at the wavelength of the maximum absorbance of Thymol blue, 434 nm, on a spreadsheet or a

Spectrophotometry (Part I & II)

PART II DATA ANALYSIS

programmable calculator, and carefully determine the slope of the best straight line fit through the data. Using this Beer's Law plot, you will determine the value of ϵ^*_{BT} .

Report the 5 absorbance readings 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).

Plot absorbance versus concentration in $\mu\text{mol/L}$ of the Thymol blue at the wavelength of the maximum absorbance of Thymol blue, 434 nm, on a spreadsheet or a programmable calculator, and carefully determine the slope of the best straight line fit through the data. Using this Beer's Law plot, you will determine the value of ϵ^*_{TT} .

Report the 5 absorbance readings 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).

Plot absorbance versus concentration in $\mu\text{mol/L}$ of the Thymol blue at the wavelength of the maximum absorbance of Bromothymol blue, 618 nm, on a spreadsheet or a programmable calculator, and carefully determine the slope of the best straight line fit through the data. Using this Beer's Law plot, you will determine the value of ϵ^*_{TB} .

Report the value of A_T , the absorbance reading you measured for the mixture at the wavelength of maximum absorbance of Thymol blue, 434 nm.

Please enter the value of A_B , the absorbance reading you measured for the mixture at the wavelength of maximum absorbance of Bromothymol blue, 618 nm.

Using the values of the effective molar absorptivities you determined from your slopes and the values of the absorption you obtained for the mixture at the spectral maxima of 434 nm and 618 nm, solve the 2x2 simultaneous linear equations for the concentrations of Bromothymol blue and Thymol blue. Report the values you calculated for the concentrations of Bromothymol blue and Thymol in the mixture.

Report the letter of the mixture sample that was assigned to you by your TA.

Determine the percent error in your analysis of the first dye. Your percent error is determined by comparing your calculated concentrations of the sample to the known values.

Determine the percent error in your analysis of the second dye. Your percent error is determined by comparing your calculated concentrations of the sample to the known values.

Plot your recorded absorbances of the mixture versus wavelength using a spreadsheet such as Excel or a programmable calculator. Be sure to turn in this graph along with the four Beer's Law plots to your TA at the beginning of the next laboratory class.

Look at the absorption spectrum of your mixture. How many peaks do you see in the spectrum?

Which species is responsible for the peaks near 436 nm and 618 nm, respectively?

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. What similarities are observed between the plots? What is the purpose of doing a scan adjustment as instructed earlier? Where does the equation $A_2 = (c_2/c_1) A_1$ come from?

Determination of Avogadro's Number

INTRODUCTION

In this experiment you will use information derived from the electrolytic decomposition of water to determine the numerical value of Avogadro's Number, N_A . As you may remember, this quantity is defined as the number of atoms in exactly twelve grams of isotopically-pure ^{12}C . Its numerical value, which must be obtained by experiment (rather than theoretically), depends upon two international agreements: the mass of one atom of ^{12}C shall be exactly 12 atomic mass units, and the amount of mass that equals exactly one gram.

A statement that the atomic mass of ^1H is 1.00780 atomic mass units means that one ^1H atom has a mass 1.00780/12 times that of the mass of one ^{12}C atom. The tabulated atomic masses of elements, which are often also loosely called their atomic weights, are dimensionless, abundance-weighted, averages of the atomic masses of the various isotopes that make up a naturally occurring macroscopic sample of the element in question.

A mole of any substance is by definition the amount of that substance which contains Avogadro's number of its constituent units. On the basis of the information in the two preceding paragraphs, it also follows that the mass of one mole of any substance in grams is numerically equal to the sum of the atomic weights of all of its elemental components. The molar mass of a substance (often termed its molecular weight) has the same numerical value both in atomic mass units and in grams/mol. Thus, the number of moles of a molecular substance can be obtained by dividing the mass of the sample by the molar mass of the molecule.

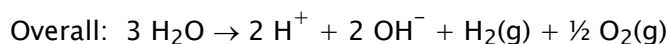
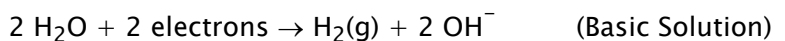
If the substance in question is a gas, then P-V-T measurements can also be used to determine the number of moles of gas in a sample if its Equation of State is known. In this experiment we will assume that the Ideal Gas Law applies.

When two chemically inert electrodes are immersed in water and a sufficiently high DC voltage is applied across them, an electric current is observed to flow and gases are evolved at both electrodes. As you undoubtedly know already, chemical tests show that one of the gases is molecular hydrogen and the other is molecular oxygen. The overall process is termed "electrolysis." (The noun "lysis" derives from the Greek word for disintegration.) A fairly straightforward way to think about the electrolysis experiment is as follows:

The "negative" pole of a battery looks negative because there are surplus electrons being made available there as a result of chemical reactions within the battery itself. If an electrode attached to the negative pole of a battery is placed in a solution containing an electron deficient species that species can take on some of the excess electrons and become reduced. This process occurs to an immeasurably small extent if only one electrode from the battery is placed in the solution.

The positive pole of a battery, on the other hand, is electron deficient. A chemical substance that is capable of surrendering electrons could thus become oxidized at an electrode attached to such a pole. Again, this process would only occur to a minute extent with an isolated electrode.

On the other hand, when both battery terminals are connected to electrodes immersed in the solution, the internal reactions within the battery will continuously replenish the electron surplus at the negative side and generate an electron deficiency at the positive side, leading to ongoing chemical processes at the two electrodes. If water is the chemical substance being electrolyzed, the following two electrode processes are:



(Note that as with all chemical reactions, the two electrode reactions are both atom-balanced and charge-balanced.)

If the solution is stirred, the H^+ and OH^- products that accumulate near the two electrodes will react with each other to make water again. The net effect of the electrolysis then will be to turn 3 moles of water into one mole of hydrogen gas and half a mole of oxygen gas for every two moles of electrons transferred around the circuit in the form of an electric current.

Since the number of moles of gas produced is directly related to the number of electrons transferred around the circuit, it looks as if all one has to do to determine Avogadro's Number is count the number of electrons that get transferred from one electrode to the other, and measure the number of moles of either hydrogen gas or oxygen gas that accompanies their transfer. The chemical product is easily quantified by collecting and measuring the volume of gas formed together with its pressure and temperature.

The number of electrons concomitantly transferred is equally easy to measure. The customary unit of electric current is called the Ampere. One Ampere of current flow corresponds to the passage of one Coulomb of electrical charge through the circuit in a one-second time interval; in other words,

$$\text{Amperes} = \text{Coulombs} / \text{Seconds}.$$

Since the Coulombic charge on an electron is known from independent physical measurements, one can determine how many electrons have been transferred in an experiment such as this one by measuring the current in Amperes, multiplying it by the time in seconds during which the current flows, and dividing the resulting product by the charge on one electron. (More precisely one should calculate the integral of current with respect to time over the course of the experiment.)

Determination of Avogadro's Number

INTRODUCTION

DATA ANALYSIS FOR PART B

At the end of the experiment you will have information in your hands concerning the volume of evolved hydrogen gas and the current vs. time profile for its evolution. In order to calculate Avogadro's number you need to compute the number of moles of evolved hydrogen and the number of electrons that were transferred while it was being evolved.

The first thing you will want to calculate is the number of electrons transferred, since it is relatively easy to obtain. Begin this calculation by determining the average value of the current along with its standard deviation. Please note that even if the observed current did not change at all over the course of the experiment, it would nevertheless have a standard deviation of at least ± 0.001 Ampere since you cannot measure it with any greater accuracy than that.

Next, multiply the average current by the total electrolysis time (in seconds) to obtain the number of Coulombs passed through the solution during the electrolysis. The resulting product must have the same relative standard deviation as the corresponding quantity for the current itself. In other words, if the current is uncertain by 5% the Coulombs passed must also be uncertain by 5%. (This statement assumes that there is no significant error in the time measurement.)

If you then divide the number of Coulombs transferred by the charge on an electron, 1.6022×10^{-19} Coulombs, you will get the number of electrons transferred. Again, the relative error in the number of electrons involved in the electrolysis must be the same as that in the current from which it was calculated.

At the end of the experiment, the buret contains hydrogen and water vapor. You can determine the amounts of each vapor that is initially present. The calculation is the same. As in the previous experiment, the pressure outside the system must be equal to the pressure inside the system. Therefore, the sum of the partial pressures of water vapor and hydrogen gas plus the pressure exerted by the column of water in the buret must be equal to the outside atmospheric pressure.

The partial pressure of hydrogen in the buret at the end of the experiment is given by the total barometric pressure minus the sum of the pressure exerted by the column of water in the buret and the vapor pressure of water. Remember that the vapor pressure of water is dependent on temperature and can be looked up in a table.

The number of moles of hydrogen produced in the electrolysis can now be calculated from the Ideal Gas Law, with "P" equal to its partial pressure and "V" equal to the total gas volume at the end of the electrolysis.

With the number of moles of hydrogen produced and the number of electrons accompanying its production both in hand, Avogadro's Number is readily calculated. The relative standard deviation of your calculated value of N_A may be taken to be the same as that of the current measurements.

Determination of Avogadro's Number
PROCEDURE

Table 1. The volume occupied by 1.0000 g water weighed in air
against stainless steel weights.

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

Table 2: Vapor Pressure of Water at Different Temperatures

Temperature (°C)	Vapor Pressure (mm Hg)	Temperature (°C)	Vapor Pressure (mm Hg)
15	12.8	23	21.1
16	13.6	24	22.4
17	14.5	25	23.8
18	15.5	26	25.2
19	16.5	27	26.7
20	17.5	28	28.3
21	18.6	29	30.0
22	19.8	30	31.8

Safety: The hydrogen gas produced is flammable; NO FLAMES in the lab. As always, exercise care when handling acids and bases. Clean up spills promptly. Wear your goggles. **Electric shock is a real possibility during this experiment. Follow the procedures carefully.**

PROCEDURE

You will work in pairs on this experiment. The actual data analyses and the written reports must be done entirely independently of your lab partner or other students.

Determination of Avogadro's Number

PROCEDURE

Stock Chemicals Used

Chemical	Maximum Amount Used
3M Sulfuric Acid	<25 mL
0.5M Potassium Sulfate	50 mL
Universal Indicator	Drops

Part A: Qualitative observations

1. Set up the DC power supply. Refer to Figure 1 on next page for terminology and set up. It is important that you make the proper connections here. If the leads are improperly connected, then the multimeter will be damaged and will not show the correct readings.

The TAs will demonstrate this set up for comparison.

2. Obtain an apparatus that consists of two funnels connected by a short length of plastic tubing, as shown in Figure 1. Attach clamps to the rubber stoppers on the stems of the two funnels and then to a support rod, producing a U-shaped configuration of the apparatus. The tops of the two funnels should be at the same height, a foot or so off the tabletop. Also, obtain two graphite (carbon) electrodes and two "clip leads." These are just fairly short lengths of insulated wire that have alligator clips attached to each end.
3. Obtain about 50 mL of 0.5 M potassium sulfate solution in a 100 mL beaker and add 15 drops of "universal indicator" in order to give the solution a fairly intense color. (Record the color you see.) We must use ionic solutions in this experiment because the conductivity of pure water is so small that it would take an impossibly long time to obtain any observable results. The ions play no part in the electrode processes. They are present to carry electric current through the solution.

The indicator used in this experiment has the behavior that its color changes in the same order as the colors in a rainbow (red, orange, yellow, green, blue, indigo, violet) as the solution changes its acid/base content by powers of ten from 10^{-4} M acid (red) through 10^{-7} M acid and/or base (a "neutral" solution) to 10^{-4} M base (violet). If you are familiar with the terminology, the indicator color changes like the rainbow as the solution pH goes from 4 to 10 in unit steps.

4. Fill the apparatus with the colored K_2SO_4 solution until the two funnels are almost full. Insert a graphite electrode into one end of each of the two clip leads, then connect the other ends of the two clip leads to the two leads coming off of the meter. The red lead coming off of the multimeter is the positive pole and the black lead coming off of the black terminal is the negative pole. Now dip the graphite electrode connected to the (+) terminal into the left-hand funnel and the (-) terminal to the right-hand funnel, keeping the metal alligator clips out of the solution.

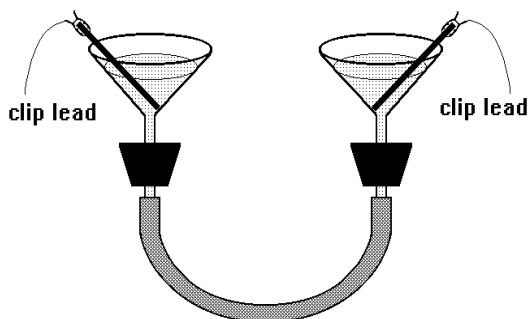


Figure 1. Funnel Apparatus with Electrodes

Make sure the power switch is in the OFF position. Turn the voltage knob all the way up, clockwise. Now turn the current knob about half way (straight up), clockwise. Now turn the power switch ON.

5. Allow the electrolysis to proceed for 10 minutes. It is important to swirl the electrode in each funnel to mix the solution, every 2-3 minutes. Turn the power OFF, and then remove the two electrodes from solution and disconnect them from the power supply. Quickly rinse the electrodes off with water from your wash bottle and store them in a clean beaker. Pipet a couple of milliliters of solution from each funnel into clean test tubes, using clean pipets. Holding the test tubes against a white background, compare the colors.
6. Very carefully unclamp one of the funnels from the supporting rod and by lowering and tipping it (using the clamp as a handle), drain all the contents of the apparatus back into the beaker from which they came.

Question A: How do you interpret the color changes in each funnel? Draw the diagram in Figure 1 and indicate what color changes occur and write balanced chemical reactions for the processes that cause the changes.

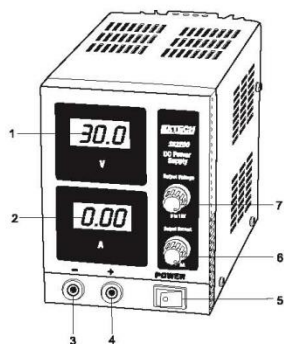
Question B: Explain the color of the solution in the beaker. Write a balanced chemical reaction for the process that caused the color change.

7. The apparatus may now be dismantled. Wash it thoroughly, rinse it with deionized water, and return it to its source.

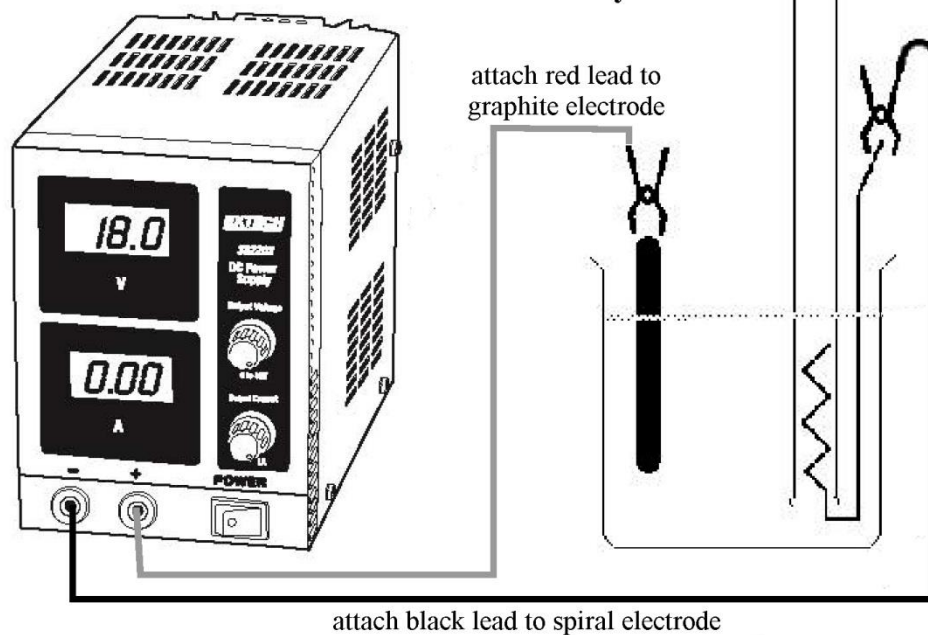
Wiring Diagram for Determination of Avogadro's Number

Power Supply / Meter Description

1. Voltage display
2. Current display
3. Negative (-) output terminal
4. Positive (+) output terminal
5. Illuminated On/Off switch
6. Current adjustment
7. Voltage adjustment



Power Supply and Electrode Assembly



rev. KBJdeCesare 21Apr09

Schematic of the Power Supply/Meter and Lead Connectivity.

Part B: Quantitative Measurements

For this part of the experiment you will need a 25 mL buret, a 150 mL beaker, a thermometer, a stainless steel spiral electrode, a graphite (carbon) electrode, two clip leads, your power supply and multimeter apparatus, and about 60 mL of 0.25 M sulfuric acid solution stored in a 150 mL beaker. You will prepare this solution yourself by diluting the 3 M stock solution supplied on the lab benches.

You will be collecting gas into an inverted buret. You will be using a 25 mL buret and will need to determine the volume of the uncalibrated portion of the buret (the head space) between the 25 mL mark and stopcock. Calibrate the buret by measuring the mass and temperature of water contained in the uncalibrated region. Deliver the volume of water into a pre-weighed dry and clean 100 mL beaker. Weigh the beaker and water and record the mass. Find the mass of water that occupies the head space. Use the conversion factors found in table 1 show above.

As you plan your procedure, remember that the water coming out of the tap is not necessarily at room temperature.

You will now electrolyze water and collect one of the evolved gases in your buret. Figure 2 shows selected parts of the assembled apparatus as it should appear at the instant the electrolysis is begun.

Determination of Avogadro's Number
PROCEDURE

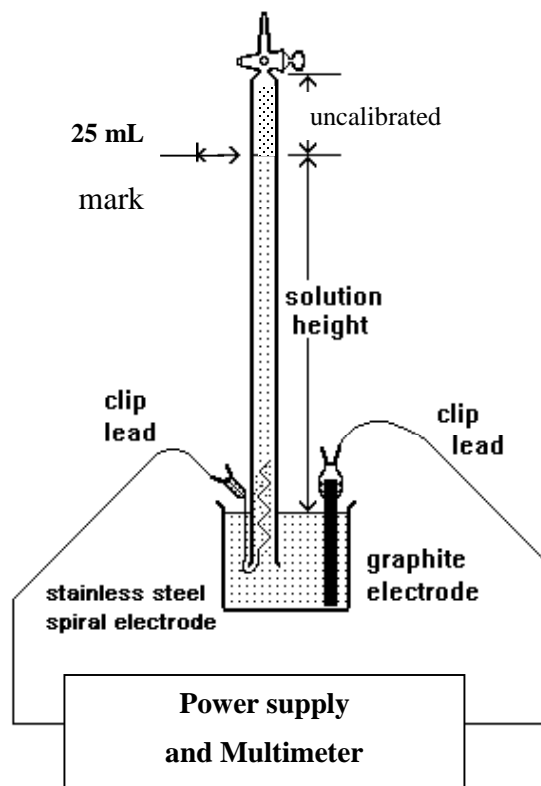


Figure 2. Electrolysis Apparatus

SAFETY: The buret will be used to catch and determine the volume of hydrogen gas that will be produced in a timed electrolysis during which the current will be monitored. The oxygen gas will be generated at a graphite electrode and will be allowed to escape into the atmosphere.

1. Invert the buret, clamp it to a support rod, and insert the stainless steel coil electrode into the buret mouth. Now lower the buret to the bottom of the beaker of dilute sulfuric acid and clamp it firmly in place. Be very careful when handling the buret from here on; the outside will have acid on it. Do not get this acid on yourself or drip it onto the floor, lab bench, neighbors and so on! Open the buret stopcock, and with a rubber bulb carefully draw the sulfuric acid solution up into the buret to top then close the stopcock, leaving no air bubbles.
2. Record the barometric pressure using the barometer mounted on one of the walls in the laboratory room. Please do not touch any of the buttons on the barometer.
3. Connect the proper lead from the multimeter to the spiral electrode. Connect the other lead from the multimeter to the graphite electrode. Be sure you are collecting the hydrogen gas at the spiral electrode! Do not put the graphite electrode into the solution in the beaker just yet. Refer to Figure 1.

Answer this question before you continue with the experiment.

Question C: To which pole of the power supply should the electrode be connected in order to collect the hydrogen gas in the buret?

4. Turn the power switch to the ON position. Adjust the voltage knob to get a reading of approximately 12.5 to 13.0 volts. Put the attached graphite electrode into the beaker as shown in Figure 1. Now adjust the current knob to get a reading of about 0.20 Amps. You will use the same setting for all part of this experiment, and there should be no further need for adjustments. Turn the power switch to the OFF position. Once again open the buret stopcock, and with a rubber bulb carefully draw the sulfuric acid solution up into the buret to top then close the stopcock, leaving no air bubbles. Turn the power on again and begin your first determination. The run time will be 10 minutes.
5. Record the meter reading every minute. Turns off the power supply after a total of 10 minutes OR if the solution in the buret drops to within 1 ml of the water line in the beaker. Once the power has been turned off, remove the graphite electrode from the solution, thereby ending the electrolysis. Store the graphite electrode in another empty beaker for future use. Wait a few minutes for all the hydrogen bubbles to rise to the top of the liquid column. Also inspect the turns of the stainless steel spiral to see if any gas bubbles are trapped there. If there are some bubbles, you can tap the buret gently and try to dislodge them. If you cannot do so, try to estimate their number and sizes and increase the gas production volume accordingly. (Recall that one buret drop is about 0.05 mL, and it is unlikely that any of the trapped gas bubbles will be that big.) When no more hydrogen bubbles are visible in the liquid column, read and record the final buret reading. Also, measure the height in millimeters of the liquid level in the buret above the liquid level in the beaker. Be sure to take and record the temperature of the solution.
6. Open the buret stopcock, allow the solution to flow back into the beaker, and close the stopcock. Swirl the beaker to homogenize the solution. You will now again use a rubber bulb to refill the buret. However, you must do this carefully so that none of the hydrogen gas in the buret escapes into the room. You want to trap all of the gas that you remove from the buret in the rubber bulb, and then vent the bulb's contents in the fume hood. While carrying the rubber bulb to the hood, use your finger to seal the bulb's opening. Squeeze and release the bulb several times in the hood to expel any hydrogen gas. It may require more than one application of the bulb to fill the buret. If this occurs, vent the bulb each time in the hood. Then, repeat Steps 3, 4 and 5 of this part of the experiment. In this repetition you should use a different (measured!!) collection time. It's up to you to decide whether to use a longer or shorter time depending on the amount of gas evolved in seven minutes. You don't want to have an evolved volume that is too small because the accuracy of your final answer depends on how big that volume is. You should not run for too long either, since as the electrolysis proceeds the solution compositions change near the electrodes (remember what happened in the two funnels in Part A of this experiment), and the current could vary somewhat erratically.

Determination of Avogadro's Number

DATA ANALYSIS

At the end of the second run, again use the rubber bulb to collect the hydrogen gas and expel it in the hood. Then drain the buret into the beaker, raise the buret up, and rinse off its outside with your wash bottle. While wearing gloves, remove the spiral electrode and place it in an empty beaker along with the graphite electrode. Rinse out the buret, wash off the electrodes with deionized water, and return everything to its proper place. Do not throw out the graphite electrodes.

Remember to perform 3 trials.

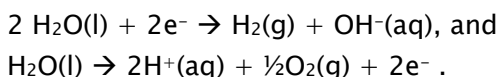
DATA ANALYSIS

Part A.

Referring to Figure 1 in the Lab manual, note any observations in your lab notebook.

For each solution in their respective funnels, decide whether it became acidic, basic, or neutral.

Decide which reaction goes with which funnel:



When you drained the solutions from the funnel apparatus and mixed them in the beaker, the blue-purple color of the right-hand funnel vanished and the solution regained its original red color. Write the reaction that occurred upon mixing together the solutions.

Part B.

What was the value of the barometric pressure in mmHg in the laboratory on the day you performed the experiment?

For each trial, what is the distance in millimeters between the liquid level in the beaker and the meniscus in the buret at the end of the hydrolysis time?

For each trial, what is the pressure in mmHg exerted by the column of liquid in your buret?

What was the temperature of the hydrolysis solution when you performed the experiment to the nearest whole degree Celsius?

What was the vapor pressure of water at the temperature at which you performed the hydrolysis?

For each trial, what is the partial pressure in atm of H₂ gas in your buret?

What was the mass in grams of water that drained out when the meniscus started just above the 25 mL mark and ended just above the stopcock?

What was the temperature of the water, to the nearest degree, that you used to determine the uncalibrated volume of your buret?

Determination of Avogadro's Number

DATA ANALYSIS

What is the volume in mL corresponding to the mass of water that you drained from the uncalibrated region near the stopcock of your buret?

What is the volume reading from the graduations on the buret at the end of the hydrolysis time for each trial?

For each trial, what is the volume of gas in your buret in mL? (Include the uncalibrated volume of your buret and the volume that results from subtracting the direct buret reading from 25.00 mL)

For each trial, what is the number of moles of hydrogen gas evolved during the hydrolysis? Use the pressure and volume of H_2 and the ideal gas law with $R = 0.0820574 \text{ L atm mol}^{-1} \text{ K}^{-1}$.

For each trial, what is the number of moles of electrons required to produce the evolved moles of hydrogen during the hydrolysis? Use the stoichiometric factor relating the moles of hydrogen to the moles of electrons.

For each trial, what is the total time in seconds of the length of the hydrolysis?

For each trial, what is the average current in amperes during the hydrolysis time?

For each trial, what is the number of electrons passing through the external circuit during the hydrolysis? Use the current law $Q = It$, where Q is the total charge in Coulombs, I is the average current in amps and t is the time in seconds, and the value of the charge of a single electron, $e = 1.6021917 \times 10^{-19} \text{ C/e}^-$.

For each trial, what is the result for Avogadro's number? Dividing the number of electrons passing through the circuit by the number of moles of electrons required for the gas evolved during the hydrolysis gives the desired result.

What is the value you calculated for the average Avogadro's number?

What is the value you calculated for the standard deviation of Avogadro's number?

What is the value you calculated for the 90% confidence limits for Avogadro's number?

What is the value for the % relative error in your determination of Avogadro's number? The accepted value is 6.022×10^{23} .

Please make some concluding remarks.

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Appendix

A) 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.*

A-1. Pre-Laboratory Preparation

Many of the Chemistry 2 laboratory experiments are intricate and use chemicals that could present a hazard if used improperly. Thus, students are required to judiciously prepare for each experiment by carefully reading the experiment and writing a Title, Purpose, Procedure (brief outline), and Data (outline) section before arriving at the laboratory. A detailed description of each section is described below under, "Writing a Laboratory Report". After preparing the laboratory notebook, students will complete the on-line pre-laboratory presentation and must pass the pre-laboratory quiz. *Any student without this preparation completed* at the beginning of the laboratory period is deemed unsafe and must leave the laboratory until the pre-laboratory write up is complete and the supervising TA is convinced that you are prepared to begin the experiment.

A-2. Data Collection

All data must be recorded *in 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.

A-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.

A-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.

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

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GENERAL EXPERIMENTAL GUIDELINES

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 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:

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.

All reports must be written in **non-erasable ink**. A date should be indicated on each report, especially in the Data section. You must prepare for each experiment by writing the Title, Purpose, and Procedure *before* coming to the laboratory. If it is not completed the student must finish it and the TA will deduct 30% of earned points from the post-laboratory exercise for first time offenders (70% for repeat offenders). Students will not be granted extra time to complete the laboratory. It is also important to organize and prepare the format of the Data section before coming to the laboratory so that you will only need to *neatly* record your data and observations during the experiment. Each section should be clearly marked with a proper heading. Your notebook should be organized and written in such a manner that another chemist could read it and repeat the experiment in precisely the same way. It is also important to complete the 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.

A-5. 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 mis-calibrated 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.

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 or samples.

The standard deviation, σ , measures how close values are clustered about the mean. The standard deviation for small samples is defined by

$$\sigma = \sqrt{\frac{\sum (x_i - \bar{x})^2}{N - 1}}$$

The smaller the value of σ the more closely packed the data is about the mean, or, in other words, the measurements are more precise.

Confidence Limits

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

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GENERAL EXPERIMENTAL GUIDELINES

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:

$$\text{Confidence Limit} = \frac{t \sigma}{\sqrt{N}}$$

where t varies with the number of observations. For the 90% confidence limits you are asked to calculate:

Number of Trials (N)	t
2	6.314
3	2.92
4	2.353
5	2.132
6	2.015

You should always report your result as the average \pm the 90% confidence limit.

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.

- Calculate the average, \bar{x} , with all data that are of high quality.
- Calculate the deviation, $|x_i - \bar{x}|$, of each *good* piece of data.
- Calculate the average of these deviations.
- 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.

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

$$Q_{\text{Data}} = \frac{\text{gap}}{\text{range}}$$

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	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!

B) On-line Pre- & Post-Laboratory Procedures

The Department of Chemistry is introducing on-line pre-& post-laboratory activities. The purpose of the pre-laboratory presentations is to aid the student in preparing for the laboratories. Each post-laboratory exercise is designed to guide you through the calculations or concepts that apply.

Prior to doing any activities, all students are required to complete the Safety Quiz online after watching the online safety videos. *Prior to coming to the laboratory class*, the pre-laboratory exercises are to be viewed and the pre-lab quiz must be completed on-line.

Read your Laboratory Manual and complete your pre-laboratory write up before viewing on-line pre-laboratory presentation.

Have laboratory notebook and calculator with you when viewing the on-line pre-laboratory presentation or completing the post-laboratory exercises. *Plan ahead*. As with any computer activity, the on-line activities may take time to complete. Do not wait until the last minute to complete any of the required on-line activities.

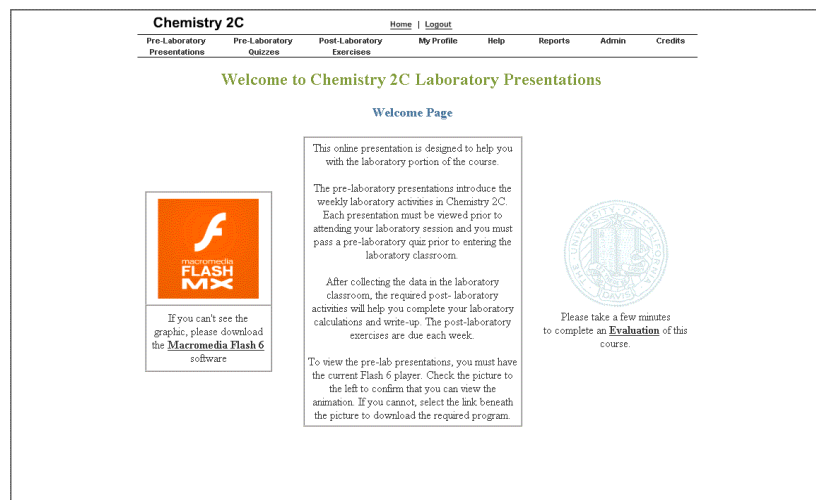
Accessing the Website

Appendix

ON-LINE PRE- & POST-LABORATORY PROCEDURES

Each time you access the On-Line Chemistry 2 Laboratory website you must do so through SmartSite and/or the web address given by your instructor.

a. The Welcome Page tests whether the Flash plug-in is working. If you do not have the correct Flash player installed. If you do not see the movie on the Welcome page, then there is no guarantee that you will be able to view all videos and slides.

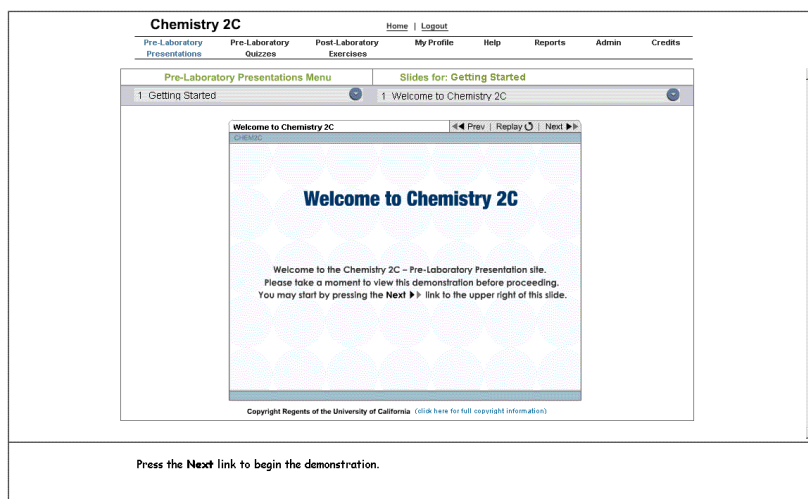


b. Click on “My Profile” to enter your personal information.

The screenshot shows the 'Edit My Personal Information' form within the Chemistry 2C website. The form has a blue header bar with the title. Below the header, there are several input fields and a dropdown menu. The fields are labeled: 'Your First name (as it appears in registration)', 'Your Last name (as it appears in registration)', 'Preferred name (name by which you are called)', 'Your UC Davis LoginID', 'Your Password (do NOT use your Kerberos password)', 'Confirm Password Change (re-type Password)', and 'Your full email address (required)'. The dropdown menu is labeled 'Change from not using CD ("No CD") to using a CD (select a path to your drive) or back.' and has options: 'No CD', 'Drive D', 'Drive E', and 'Drive F'. At the bottom of the form are two buttons: 'Submit Changes' and 'Reset'. Below the form is a link 'Return to My Profile Menu' and a small copyright notice at the very bottom.

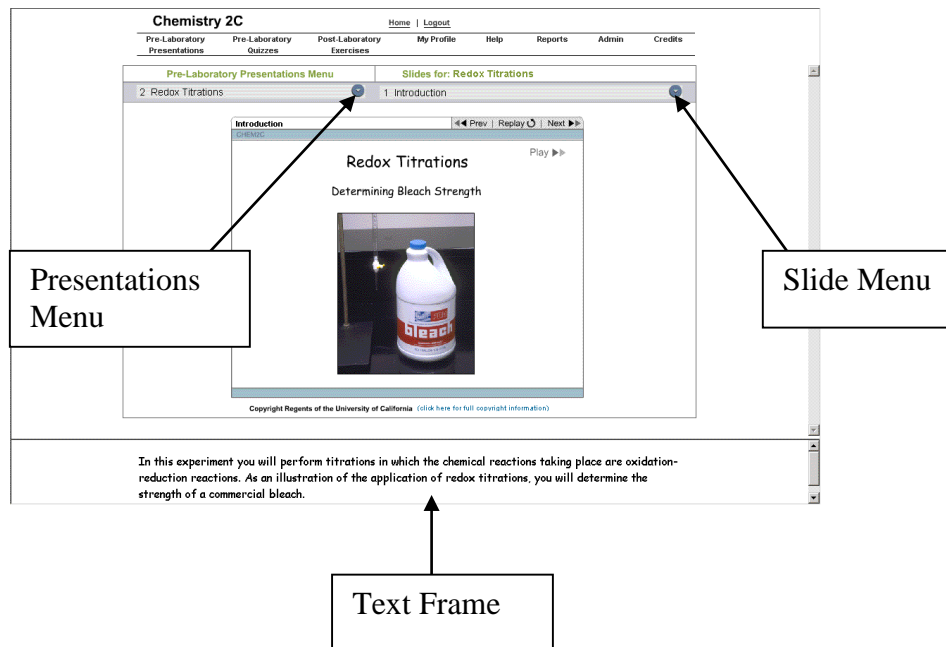
B-1. Viewing the Pre-laboratory Presentations.

a. If you click on “Pre-Laboratory Presentations,” it will take you to the Pre-Laboratory Presentation screen as seen below. There is a brief tutorial in the “Getting Started” Presentation.



NOTE: If you run into difficulty with any of these steps, please contact your lab TA.

b. Sequentially view the slides by either clicking on “Prev” and “Next” buttons, or view any slide in the presentation by selecting it in the Slide Menu. Note you may review any slide at any time.



c. The entire text for each slide may be viewed by moving the slider directly to the right of the text frame.

d. Audio is provided but not essential. All the information is conveyed in the text and the main frame.

Appendix

ON-LINE PRE- & POST-LABORATORY PROCEDURES

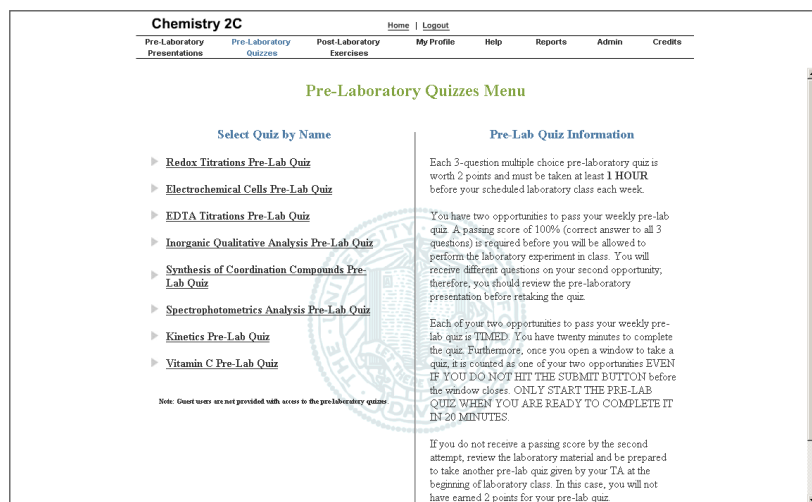
B-2. Taking the Pre-laboratory Quiz

After viewing the lab session, go back to the Chemistry 2 Laboratory Presentation Home Page by clicking at the top of the page.

a. Click on pre-laboratory quizzes. Choose the appropriate laboratory quiz. Each pre-lab quiz must be completed *at least 1 hour* prior to attending your scheduled lab class. A passing score of 100% (correct answers to all three questions) is required before you will be allowed to perform the laboratory experiment. If you fail the quiz on the first attempt, you may take the quiz a second time. Because the questions are chosen randomly, you may receive different questions on your second attempt, so it is a good idea to review the pre-lab session prior to your second attempt. You may also view the laboratory session while you are taking the prelaboratory quiz. If you fail to pass the quiz on a second attempt, review the laboratory material again and be prepared to take another prelaboratory quiz at the beginning of laboratory class given by your TA but you will not receive any points.

b. *Pre-lab quizzes are timed quizzes.* You have twenty minutes to take the quiz. Furthermore, once you open a window to take a quiz, it will be counted as one of your two attempts *even if you do not hit the submit button* before closing the window. *Only start the pre-lab quiz when you are ready to take it.*

c. In order to receive your 2 points for the prelaboratory quiz you must complete it successfully at least *1 hour* before your laboratory class is to begin.



B-3. Completing the Post-Laboratory Exercises.

You will need to complete all the on-line post-laboratory exercises for each lab in order to receive credit for the laboratory portion of the course.

In the post-laboratory exercises, you will be asked to enter your data and the results from your calculations. For your data entries, the post-lab exercises are designed to check that your data is sensible. For example, if you are asked to weigh approximately 3 g of a substance, the program will check to see if your data entry falls within a range such as 1 - 6 g.

For your calculation entries, the program is designed to verify that your calculation is correct based on your previously entered data. The program also allows for rounding differences. For example, if the program is expecting the entry, 0.234, based on your data, then a value in a range of 0.232 – 0.236 may be accepted.

There are also multiple-choice questions and free response questions posed in the post-lab exercises. An on-line text box will be provided for you to write any concluding remarks discussing and explaining your experimental results.

a. Click on post-laboratory exercises. Choose the appropriate laboratory exercise and follow the instructions. See below.

The image shows two screenshots of a web-based laboratory exercise interface. The top screenshot is the 'Post-Laboratory Exercises Menu' for 'Chemistry 2C'. It features a navigation bar with links: Home, Logout, Pre-Laboratory Presentations, Pre-Laboratory Quizzes, Post-Laboratory Exercises (selected), My Profile, Help, Reports, Admin, and Credits. The menu lists several exercises: Redox Titrations Post-Lab, Electrochemical Cells Post-Lab, Nomenclature Quiz, EDTA Post-Lab, Qualitative Analysis (Week 1) Post-Lab, Qualitative Analysis (Week 2) Post-Lab, Synthesis Post-Lab, Spectroscopy Post-Lab, Kinetics Post-Lab, and Vitamin C Post-Lab. A 'Post-Laboratory Exercise Information' section provides instructions: 'Each post-laboratory exercise is designed to guide you through the calculations or concepts that apply. Your calculations are verified using your data entries.' It also includes advice on handling incorrect responses, the importance of careful entries, and the need to keep a detailed record of data. A note at the bottom states: 'Note: Count scores are not provided with access to the post-laboratory exercise.' The bottom screenshot shows the 'Redox Titrations Post-Lab' exercise. It has a blue header and a green bar indicating 'Previous Question Score/Possible Points: No score possible' and 'Total Score So Far/Total Exercise Points: 0/40'. The main text area contains instructions about hyphenated numbers on a locker (e.g., '0435-6-24') and a prompt to log off. At the bottom, there are 'Submit Answer/Continue' and 'Quit Post-Lab' buttons. Below the buttons is a 'Post-Lab Data Summary' section with a note: 'Note: some questions will display a variable like "nCount" or "SInput" instead of an actual number in the data summary.' and a final instruction: 'As you complete this Post Lab Exercise, your previous Answers will be displayed here for reference.'

b. You will need to have your laboratory notebook and a calculator or spreadsheet program to complete the exercises. You should keep a detailed record of your data entries and the resulting calculations in your laboratory notebook. You may need to reference this material when discussing a calculation with a TA.

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ON-LINE PRE- & POST-LABORATORY PROCEDURES

c. As you proceed through your post-lab exercise, a scroll down window appears at the bottom of the screen. This summary is the post-lab data summary, and it contains your accepted entries and the number of points awarded for each question. You may refer to this summary to verify the values you entered that are used in subsequent calculations.

d. When asked to collect data for multiple trials, you must have data for at least 3 trials to complete the post-lab exercises. The single exception to this is Part III of the Vitamin C laboratory. When entering data or calculated values, do not include unit symbols.

e. In many cases, you will not be able to proceed to the next question until you have correctly answered the previous question. Some hints are provided for the first few incorrect responses. If you are unable to proceed after repeated attempts to enter a correct response, please contact your TA.

f. Be careful and deliberate about your entries. Once you proceed to the next question, you cannot go back and change your answer to a previous question.

g. In contrast to the pre-lab quizzes, you may exit the post-laboratory exercise at any time and re-enter as many times as you wish. Upon re-entry, the program will begin with the same question that you were answering when you exited. Points are not awarded until you click the submit button.

Scoring Scheme

The first line of text on each question contains a terse notation describing the scoring for that question. The notation used and an explanation of each is provided below:

1. Data Entry – No Scoring

Simply enter your experimental value. The program will verify that your entry is within the expected range for the experiment, but no awarding of points is involved.

2. Scoring Scheme: 2–1

These are typically questions that have only two alternative answers. If you select the correct answer, you will receive two points. If you select the incorrect answer, you will receive one point for completing the question and you will be informed of the correct answer.

3. Scoring Scheme: 3-2-1-1

These are typically multiple-choice questions with three alternatives. If you select the correct answer on the first try, you will receive three points. The possible points earned are then reduced by one point on each try and a hint is provided. You will receive a minimum of one point if you answer correctly on the third or subsequent tries.

4. Scoring Scheme: 3-3-2-1

These are typically questions that require you to do calculations based upon previously entered experimental data, but may also be multiple choice questions with 4 or more alternatives. If you respond correctly on either of the first two tries, you will receive three points. The possible score is reduced by one point for each of the next two tries and remains one point for a correct response on any subsequent try.

5. Free Response (1 or 2 points possible)

Some of the laboratories contain questions where you will write your answer in a text box. The point value for each question will be indicated. Your TA will read your responses and award you your points accordingly. Your points for these questions will appear in your on-line score sheet.

6. Analysis (1 to 5 points possible)

In some of the laboratories, you will analyze a sample of unknown content. In the Redox and EDTA laboratories you will find a mass percent and in the Qualitative Analysis laboratory you will be identifying the metal ions present in a mixture. In these three laboratories, you will be awarded 1 to 5 points for accuracy. In order for the on-line program to identify which sample you were assigned to analyze, you will need to enter your locker series number.

Due Date/ Late Submission of Post-lab Exercise

The post-laboratory exercises must be completed by the next normally scheduled laboratory meeting. The last post-laboratory exercise is due **the last day of instruction**. Each post lab exercise has a date/time stamp to indicate the date and time of completion. **Late submission of your post lab exercise will be met with a 5-point deduction for every calendar day it is late.**

NOTE: If you run into difficulty with any of your post-laboratory entries, please contact your TA.

Appendix

LATE REPORTS & MAKE-UP POLICY

C) Late Reports & Make-Up Policy

C-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.***

C-2. Laboratory Make-Up Policy

Students must attend the laboratory class for the section in which they are enrolled. If a student misses a laboratory class with an excused absence, ***it must be made up before the end of the following week of laboratory.*** No further opportunity for make-up will be provided to the student who fails to make up the lab by the following week. No make ups for unexcused absences. ***If a student misses the last lab of the quarter, it must be made up immediately.*** Typically, laboratory classes end one or two days before the end of the quarter. ***No laboratory make-ups will be offered after one week from the scheduled date of the lab.*** Excused absences include an extended illness or family emergency. Bring proof to your TA or head TA immediately upon return. If you cannot present this proof or have an unexcused absence, you may receive a failing grade in the course.

C-3. Laboratory Make-up Procedure

You are required to complete all labs in order to pass the course and it is your responsibility to make up any missed labs promptly. Failure to make up a lab may result in a ***failing grade*** for the course.

If you miss a lab, you must make it up by attending another scheduled laboratory section. 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.***

C-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.

D) Common Laboratory Procedures

D-1. Handling Solids

General Guidelines for Handling Solids

- Use a **clean** spatula or scoopula to transfer solid from bottles. Never use a contaminated spatula.
- Never return unused solid to the reagent bottle. To eliminate waste, avoid removing more solid from a bottle than is necessary.
- Never discard chemicals in the trashcan. Follow waste disposal procedures outlined in the Laboratory Manual.

Quantitative Transfer

Quantitative transfer refers to 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 weigh boats rather than weighing paper, but the procedure is essentially the same.

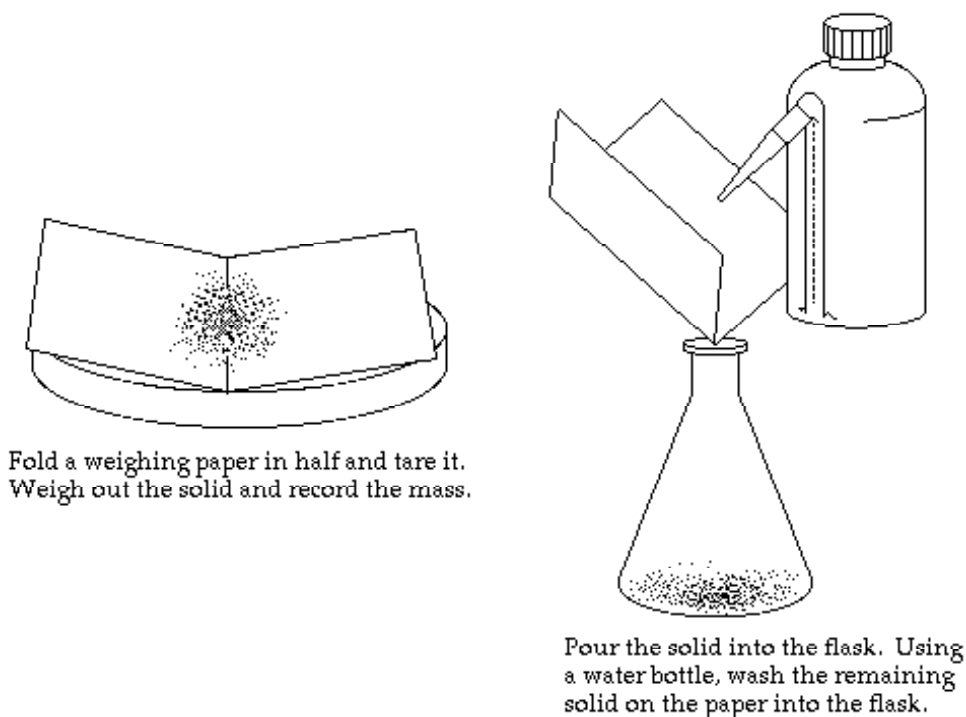


Figure 1 Quantitative Transfer of Solids

Appendix

COMMON LABORATORY PROCEDURES

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.



- Keep the desiccator closed at all times.

The desiccant will absorb moisture in the air extremely rapidly.

- 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.

D-2. Handling Liquids

Drawing Solutions from a Reagent Bottle

Most reagent bottles in your laboratory have a small test tube holder 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 pipette 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. May result in chemical splashes.

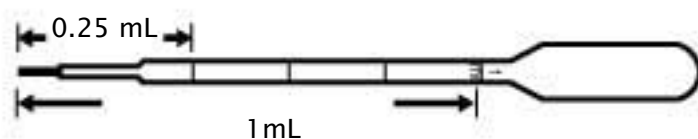
Always put full dispo pipet in a test tube when carrying it to another part of the lab.

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 1 mL of solution into an empty dispo pipet:

1. Squeeze the bulb to remove some air from the dispo pipet.
2. Submerge the tip of the dispo pipet in the solution.
3. Slowly release the pressure on the bulb and draw solution to the 1 mL mark.
4. Without releasing pressure on the bulb, steadily remove the dispo pipet from the solution.



Transferring Liquid

- 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.

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Pour the liquid slowly and carefully to avoid spillage. You may find the use of a glass rod helpful, as shown below.

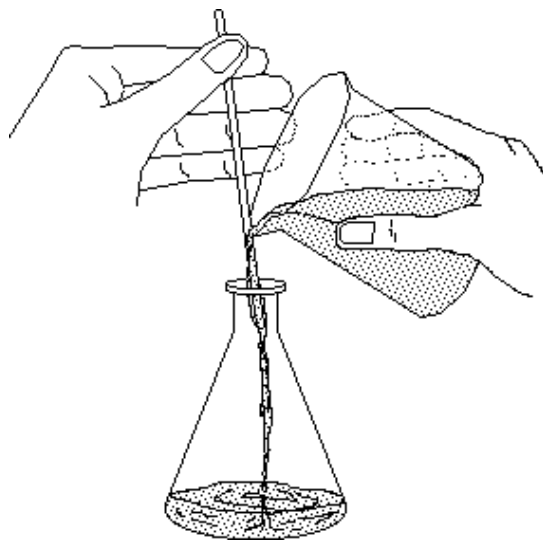


Figure 2. Liquid Transfer with Glass Stir Rod

- 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.

D-3. Capping a Flask with Parafilm

During many experiments you will have to cap a flask to protect the contents from contamination. Figure 4 illustrates the proper method using Parafilm.

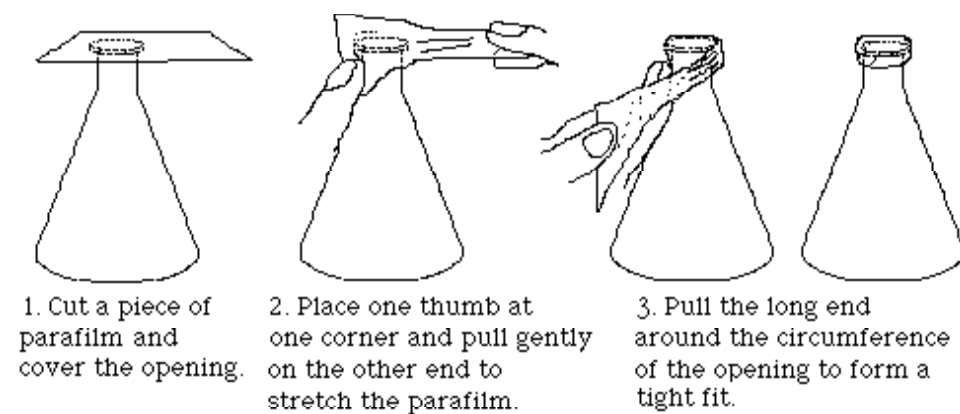


Figure 3 Capping A Flask

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. **Error! Reference source not found.** 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.

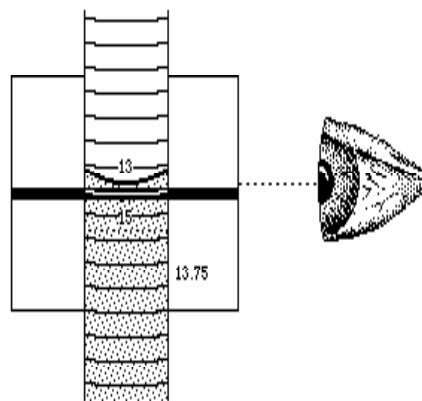


Figure 4 Reading the Meniscus

D-4. 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.

Care and Maintenance of Laboratory Glassware

- Always examine the glassware for chips and scratches before use. Damage glassware may break during normal usage or cause bodily injuries.
- Never place glassware near the edge of lab bench. Keep the work area clean and organized to prevent accidents and chemical spills.
- Clean broken glassware must be disposed of inside the designated Glass Disposal Box. If box is full, ask the dispensary for a new one.
- Clean all glassware with water. Make sure to rinse the glassware with DI water as a final step.
- Never heat glassware to dryness. Add cold water with your 250 mL water squeeze bottle when needed.
- Never place a heated beaker in an ice bath, or vice versa. Allow the glassware to warm up or cool down gradually.

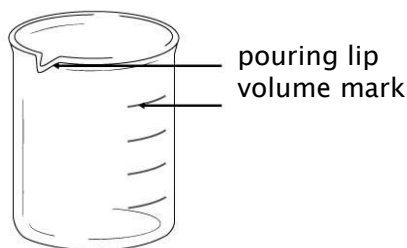
Appendix

COMMON LABORATORY PROCEDURES

- Never carry lab ware by the neck or cap. Always hold lab ware from the bottom and the side.
- 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”).

Beakers

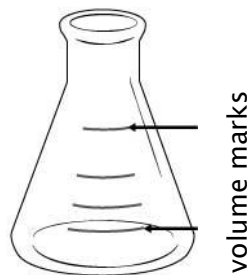
Beakers can be used in the laboratory to estimate volume, storing liquids temporarily, and carry out certain reactions.



- Always hold beakers from the bottom or the side. Never hold a beaker by the rim.
- All beakers in the Chemistry 2 laboratories have a pouring lip to make pouring solutions easier.
- All beakers in the Chemistry 2 laboratories have marks for estimating the volume. As noted on the glassware, there is a $\pm 5\%$ error for the largest volume mark.
- Place a 100 mm watch glass on top of beaker when boiling water to speed up the process.
- 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!*

Erlenmeyer Flasks

Erlenmeyer flasks can be used in the laboratory to estimate volume, storing liquids temporarily, and carry out certain reactions.

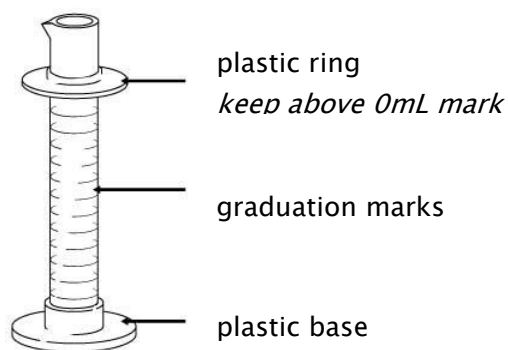


- All Erlenmeyer flasks in the Chemistry 2 laboratories have marks for estimating the volume. As noted on the glassware, there is a $\pm 5\%$ error for the largest volume mark.
- If needed, write labels in the frosted areas on the beaker with graphite pencils (a.k.a. “lead” pencils). *Do not use wax pencil or pen!*

Graduated Cylinder

Graduated cylinders are used to measure a small volume of liquid with more precision than beakers and Erlenmeyer flasks.

- 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 when the glassware is knocked over. Make sure the plastic ring is placed near the top of the cylinder.

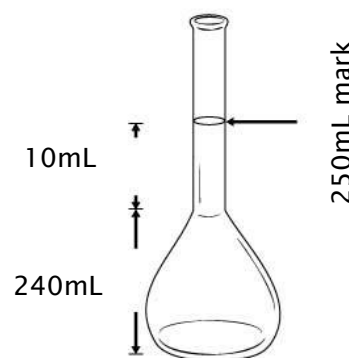


- To quickly measure out a specific amount of water, fill your 250 mL water squeeze bottle with DI water and squeeze the desired amount of water into the graduated cylinder.

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.

- 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.
- To fill a volumetric flask to the mark, quickly fill the flask to 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.
- Never use glass pipets or dispo pipets with the volumetric flasks. Pipets will become stuck inside the flasks.



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,

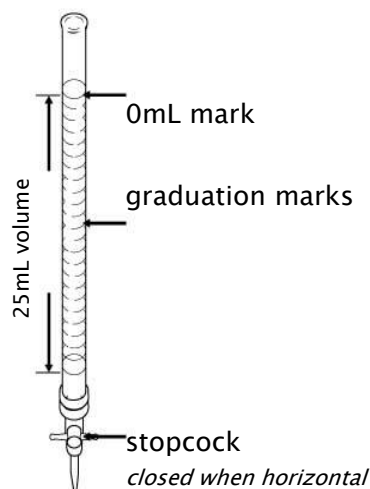
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burets are calibrated to measure the liquid *delivered* from the glassware. In the Chemistry 2 labs, the buret is mostly used for titrations.

Filling the buret with DI water

1. Always remove the buret and hold it below your eye level when filling the buret.
2. Check to make sure the stopcock is in the closed position.
3. Always use a small beaker (100 mL or 150 mL). For a 25 mL buret, pour 30-40 mL of DI water into the beaker.
4. Hold the buret slightly below the 0mL mark with one hand; slowly pour into the buret the solution from the beaker in your other hand. Stop before the liquid level reaches 0 mL.



Cleaning the buret

1. To clean a buret, fill it to half way with DI water.
2. 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.

Conditioning the buret

You should always *condition* your buret with your working solution before using it.

1. Clean the buret with DI water.
2. 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.
3. 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.
4. Pour the remaining solution in the buret into an appropriate waste container.

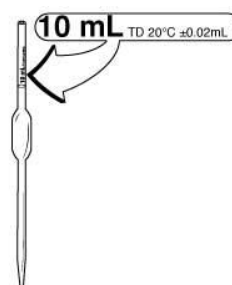
Dispensing solution from the buret

1. 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*.
2. 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 *final reading* is the volume dispensed.
3. To dispense in small quantities, quickly turn the stopcock clockwise exactly 180 degrees. Repeat as needed.

Volumetric Pipet

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

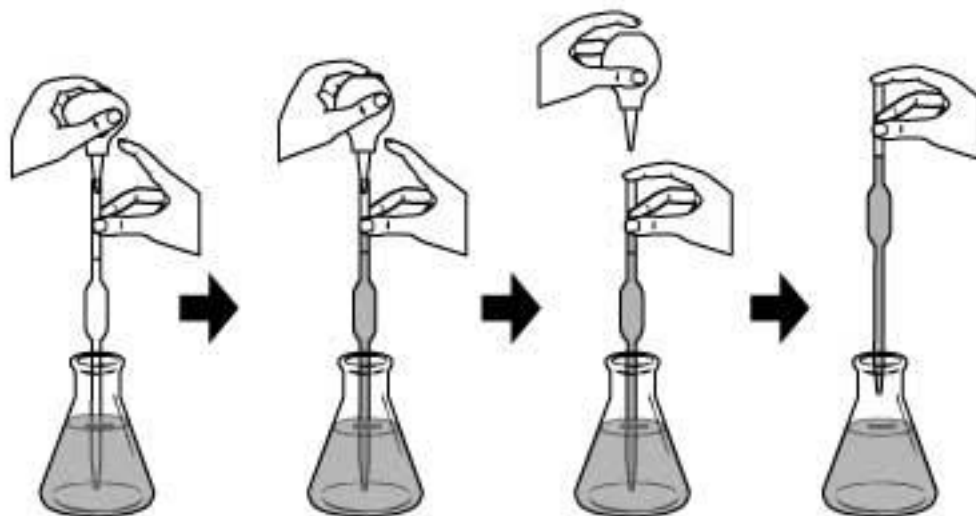
- The volume of liquid each pipet is designed to deliver is labeled on the glassware. Use the volumetric pipet *only* when you need to deliver the exact amount of solution with precision.
- There is a bottle of volumetric pipet cleaning solution in each laboratory. Draw the cleaning solution into the pipet with a pipet bulb and dispel the solution
- 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.
- Follow the illustration below 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.

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1. To begin:

- With your dominant hand, hold the conditioned pipet vertical and the pointed end downward inside the container of your working solution. Place your hand near the top of the pipet and keep your 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 tip inside 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 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.

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COMMON LABORATORY PROCEDURES

D-5. Using the Balance

A balance is used to measure the mass of an object. There are 4 analytic 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.

On/Off Switching

- To turn on the balance, remove all load from the weighing pan and press the **On** button.
- To turn off the balance, press and hold the **Off** key for 2 seconds.

Simple Weighing

Open one of the draft shield sliding door. 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** 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.

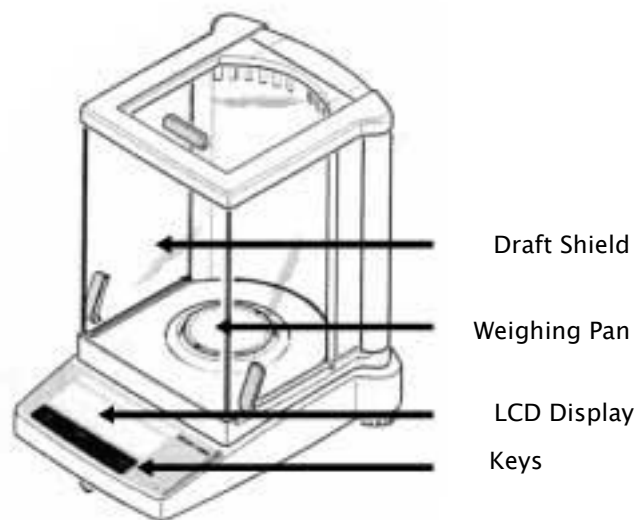


Figure 5. The Analytical Balance

Notice

Always use weighing boats when weighing solids to protect the balance. To do this simply place the plastic weighing boat on the balance pan and be sure it is not touching the side.

Always use the balance with extreme care, as it is very expensive.

Taring

To measure the mass of sample inside a container, perform the following procedures:

1. Place the empty container (e.g. a weighing boat) on the balance.
2. Press the **0/T** key briefly. The display should read 0.000 g.
3. Add the sample to the container. Read the displayed mass to 0.0001 g.

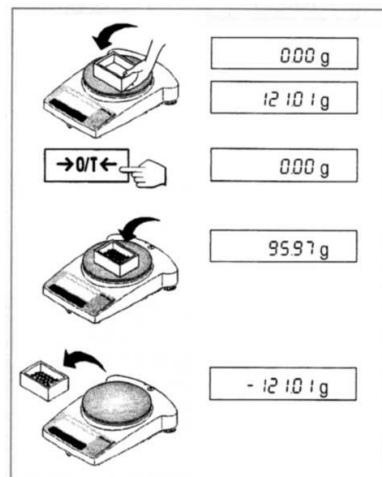


Figure 6 Taring

Weighing by Difference

To measure the mass of a sample by difference

1. Clear the weighing pan. Press **0/T**. The reading should be 0.000 g.
2. Place the container with the sample on the balance. Record the mass.
3. Remove a portion of the sample from the container.
4. The difference between the two readings is the mass of the removed portion of the sample.

D-6. 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.

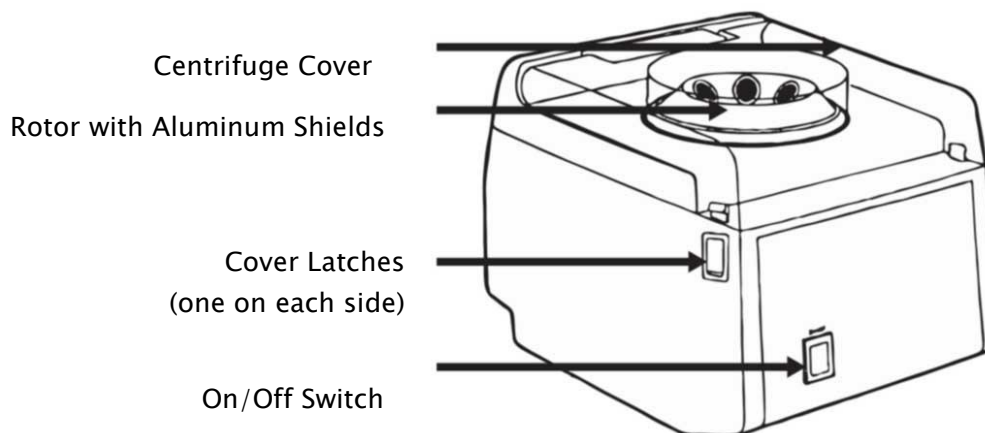


Figure 7 The Centrifuge

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Procedure

1. Always load centrifuge tubes of about equal weight. Fill another centrifuge tube with water to equal weight to balance.
2. 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).
3. 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.
4. Close the cover. Lock both sides securely into the latches.
5. 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.

Safety Precautions

- Operate the centrifuge only when the cover is securely closed.
- Never open the cover when the centrifuge is running.
- Always balance the tubes before loading. Only spin 2, 4, or 6 tubes.
- Never spin 1, 3, or 5 tubes.
- Turn off the machine immediately if there are signs that the load is unbalanced.
- Never open the cover before the rotor comes to a **complete** stop.
- Never stop the rotor with your hand. Serious injury may result.

D-7. 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.

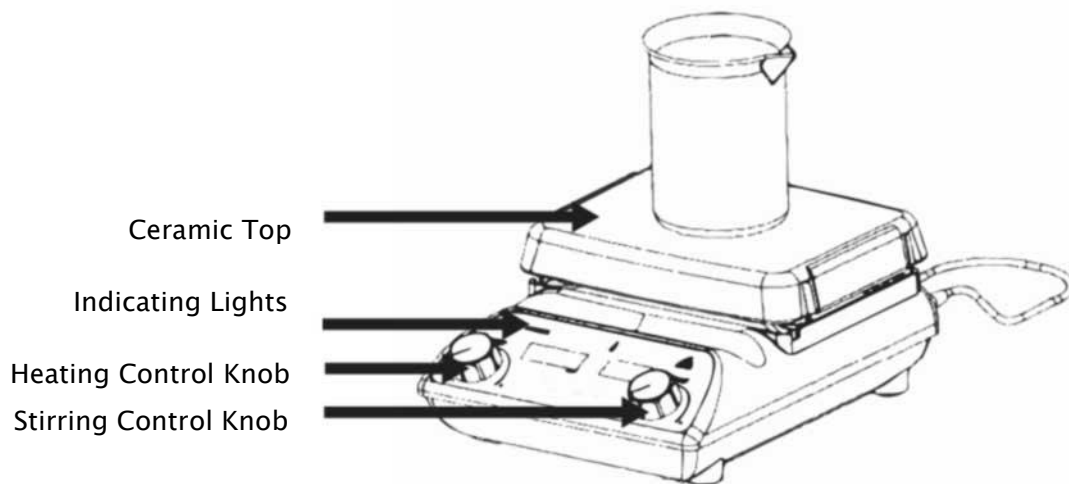


Figure 8 The Hotplate/Stirrer

Features

- 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.
- 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.

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Safety Precautions

- Keep the power cord away from the heating surface. The cord may melt and cause an electrical hazard.
- Do not hit the top with heavy objects. It may break if impacted.
- Do not heat volatile or flammable materials.
- Do not operate near volatile or flammable materials.

The hot plate must not be used during these experiments:

- 2A. Determination of Avogadro's Number
 - 2B. Colligative Properties
- 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.

- Never use a container larger than the top plate.
- Never boil a solution to dryness.

D-8. 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.

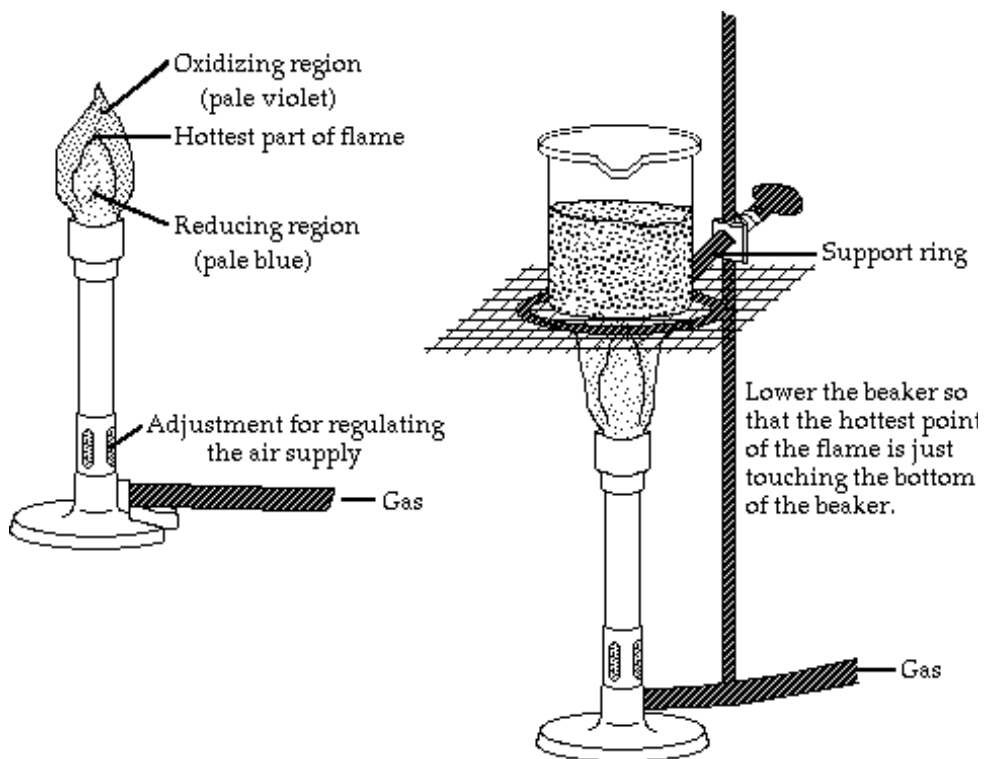


Figure 9 The Bunsen Burner

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!

D-9. 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.

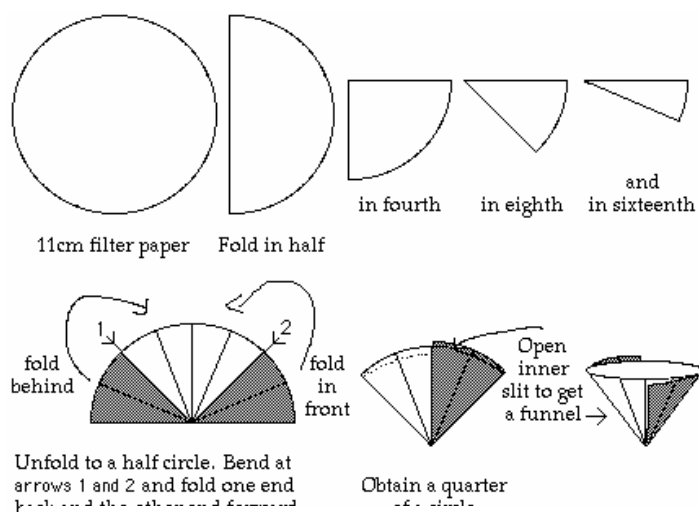


Figure 10 Fluting the Filter Paper

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.

D-10. pH Meter Operating Instructions



A. Preparing the pH meter

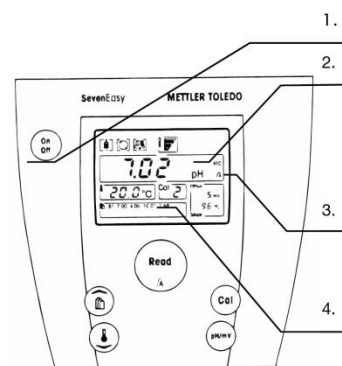
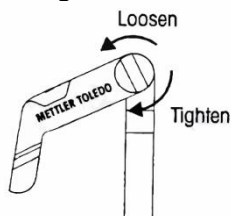
A-1. Turn on the pH meter

A-2. Meter must be in pH mode. If in mV mode, press **pH/mV** button.

A-3. Make sure pH meter is showing \bar{A} . If not shown, press and hold **Read** button for 2 seconds.

A-4. Lower left window must show **B1 7.00 4.00 10.00 1.68**. If not, ask your TA to adjust the setting.

A-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.



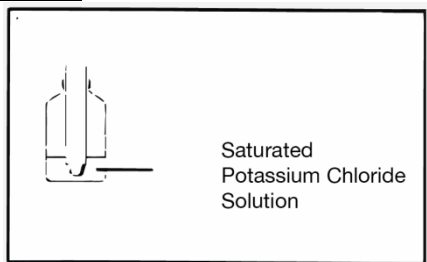
Caution

Do NOT place test tubes on electrode stand!

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COMMON LABORATORY PROCEDURES

A-6. Do NOT let electrode dry out. Always store electrode in sat. KCl solution when not in use.



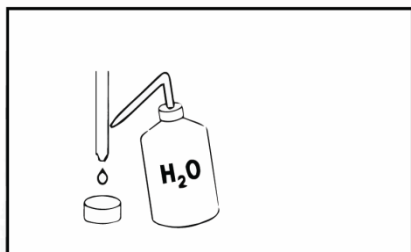
B. Calibrating the pH meter

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

B-1. Rinse the electrode with DI water.

B-2. Blot dry with Kimwipe.

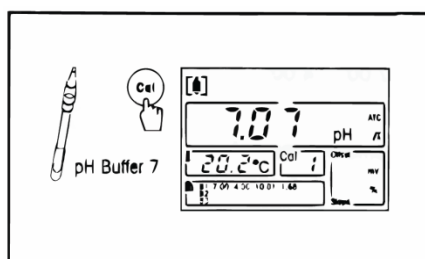
WARNING: Do NOT rub the electrode with Kimwipe. Rubbing the electrode may build up static charge and damage the electrode.



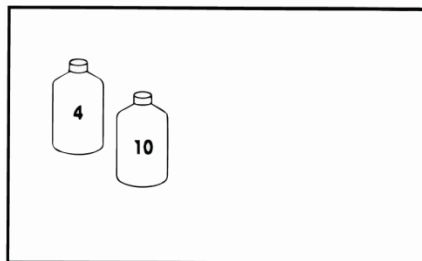
B-3. Place electrode in pH 7 buffer standard (yellow).

B-4. Press the **Cal** button.

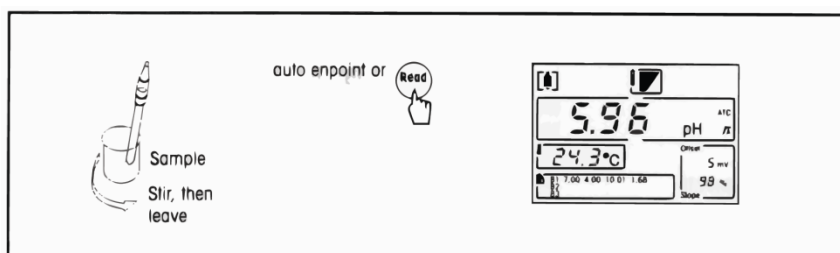
B-5. Wait for the display to stop blinking.



B-6. Repeat step B-1 to B-5 with the pH 4 buffer standard (red) and with the pH 10 buffer standard (blue).



C. Measure the pH of sample



C-1. After calibration, place electrode in sample solution and press **Read**.

C-2. Wait for the reading to stabilize.

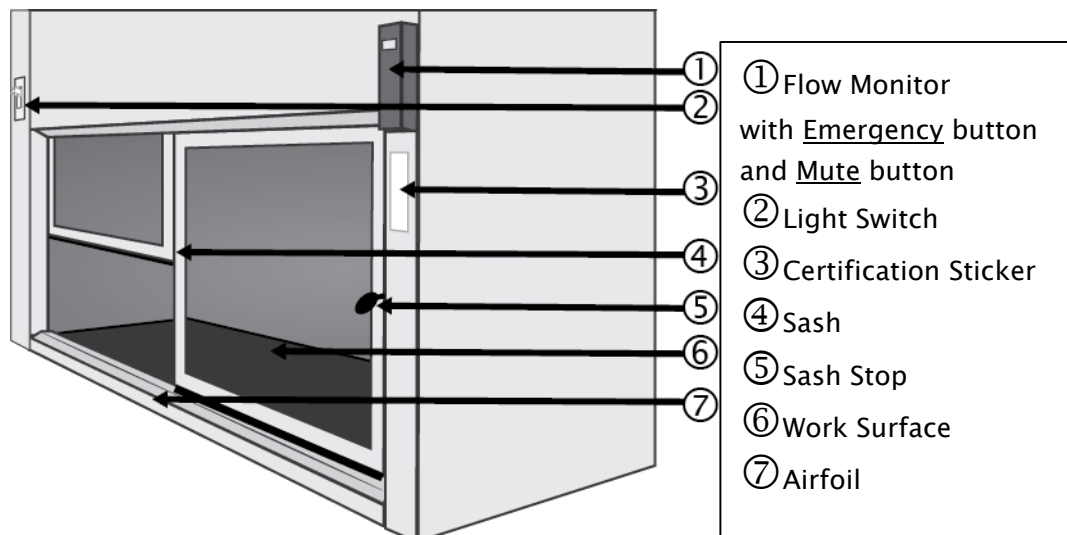
Appendix

COMMON LABORATORY PROCEDURES

D-11. Fume Hood Use and Safety

The fume hoods in the laboratory protect personnel from hazardous materials and inhalation of toxic materials.

Features of the Fume Hood



Before Using the Fume Hood

- Check the certification sticker (③). The Fume Hood is tested and certified every year.
- 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.

- Turn on light switch (②).

Working With the Fume Hood

- Lift the sash up slowly about 12 inches. Never raise the sash above the sash stop(⑤).
- Always place lab equipment at least six inches away from the edge and inside the fume hood as much as possible.
- Do not rest body parts on the edge or the Airfoil (⑦).
- Do not place glassware or chemicals on the Airfoil (⑦).
- 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!**

- When increased airflow is needed, press the Emergency button and the Mute button.
- **Clean up spills immediately.**
- **Cap all containers immediately.**

- Turn off Emergency mode, close hood sash all the way at the end of lab.

Fume Hood Usage in Chemistry 2 Laboratory

- **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.

- Many hazardous chemicals are kept in the fume hood. Never remove these containers unless specifically directed by the Laboratory Manual.
- All Hazardous Waste containers for the Chemistry 2 course are kept in the fume hood.

Fume Hood Emissions

- A. Minimize fume hood emissions to protect the environment and air quality.
- B. Never evaporate waste in the fume hood.
- C. Minimize use of volatile liquids. Close and seal after using.

If you have questions, contact your TA or safety coordinator.

Appendix

SAFETY IN THE CHEMISTRY 2 LABORATORIES

E) 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 **Person Protective Equipment (PPE)**.

Special Medication Condition

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

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.

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 with water for 15 minutes with water. Seek medical attention.

- **Drench 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.

For small chemical spills on your arms and hands, use the drench hose. Wash affected area with 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.

When the safety shower is used in the Science Lab building, all other students must evacuate the room. The TA must dial 911 and inform the Fire Department that the safety shower is used.

- **Fire Alarm Box**

The fire alarm boxes in the Science Lab building are located in the hallway.

Fire Alarm and Evacuation

When the Fire Alarm sounds, stop all reactions and turn off all sources of ignition (e.g. hot plates, Bunsen burner). Exit the building calmly according to the evacuation plan and go to the designated area (i.e. the mustering point). Your TA will take roll call to make sure you are safely out of the building.

Do not leave the designated area unless instructed by your TA or emergency response personnel.

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.

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 instructed by the TA or the laboratory staff.

Follow all instructions posted in the laboratory.

Practice Good Housekeeping

- Keep work area organized. Don't put glassware on edges where they may fall off.

Appendix

SAFETY IN THE CHEMISTRY 2 LABORATORIES

- Cap all bottles and close all drawers as soon as you are done using them.
- Clean up all spills and broken glassware immediately.

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.

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 sleeve shirt, 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:

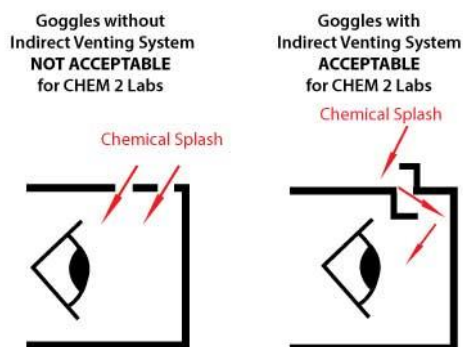
No skin exposure from the neck down to the feet in the laboratory.

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.

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 extend to the wrists. Do not wear a lab coat that's too small or too big for you.
- Keep your lab coat buttoned at all times.

Appendix

SAFETY IN THE CHEMISTRY 2 LABORATORIES

Gloves

You will be provided with disposable nitrile gloves in lab for your 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.**

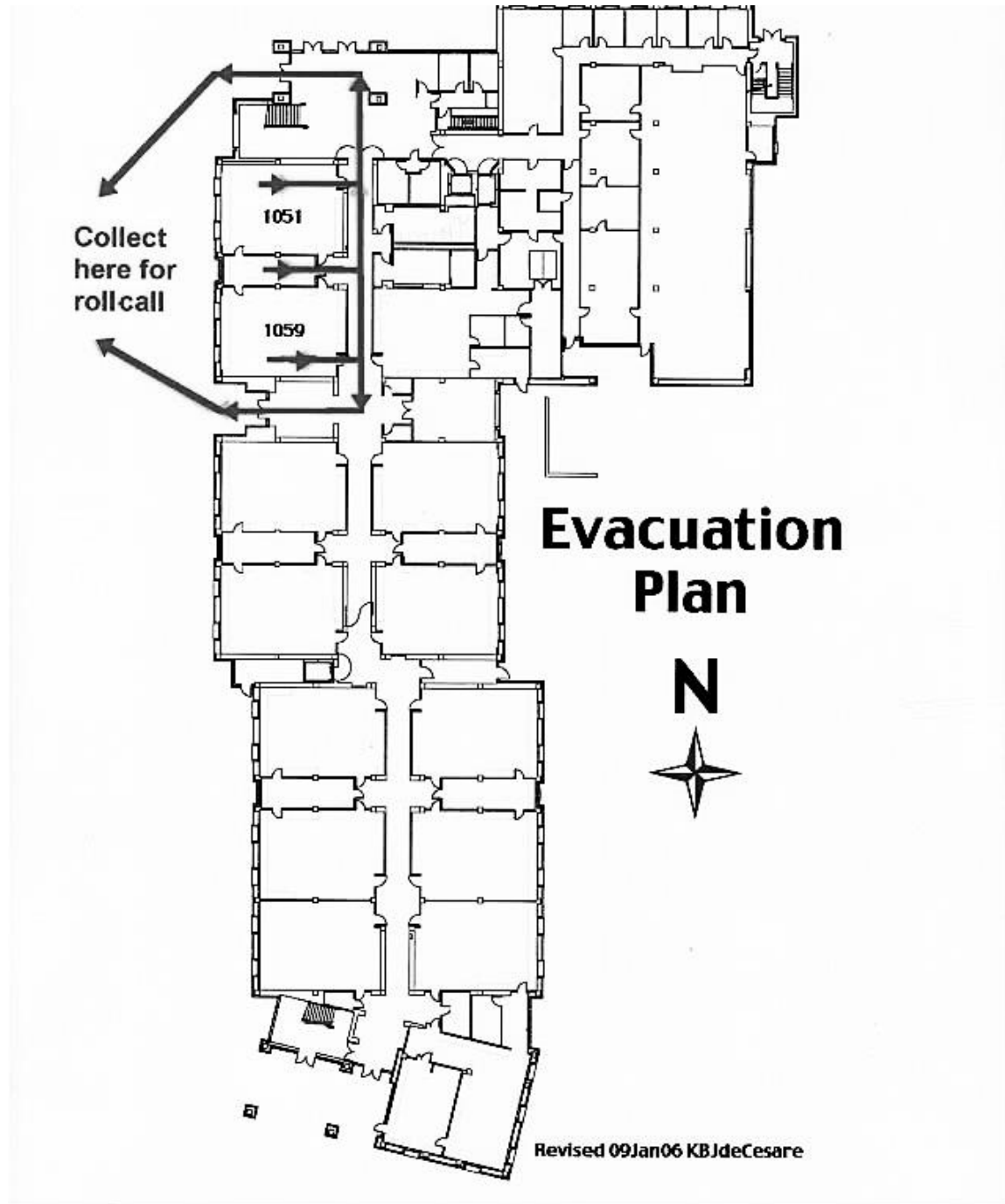
Allergy

- If you are allergic to nitrile gloves, contact your TA and the laboratory staff. You will be provided with hypoallergenic lab gloves.

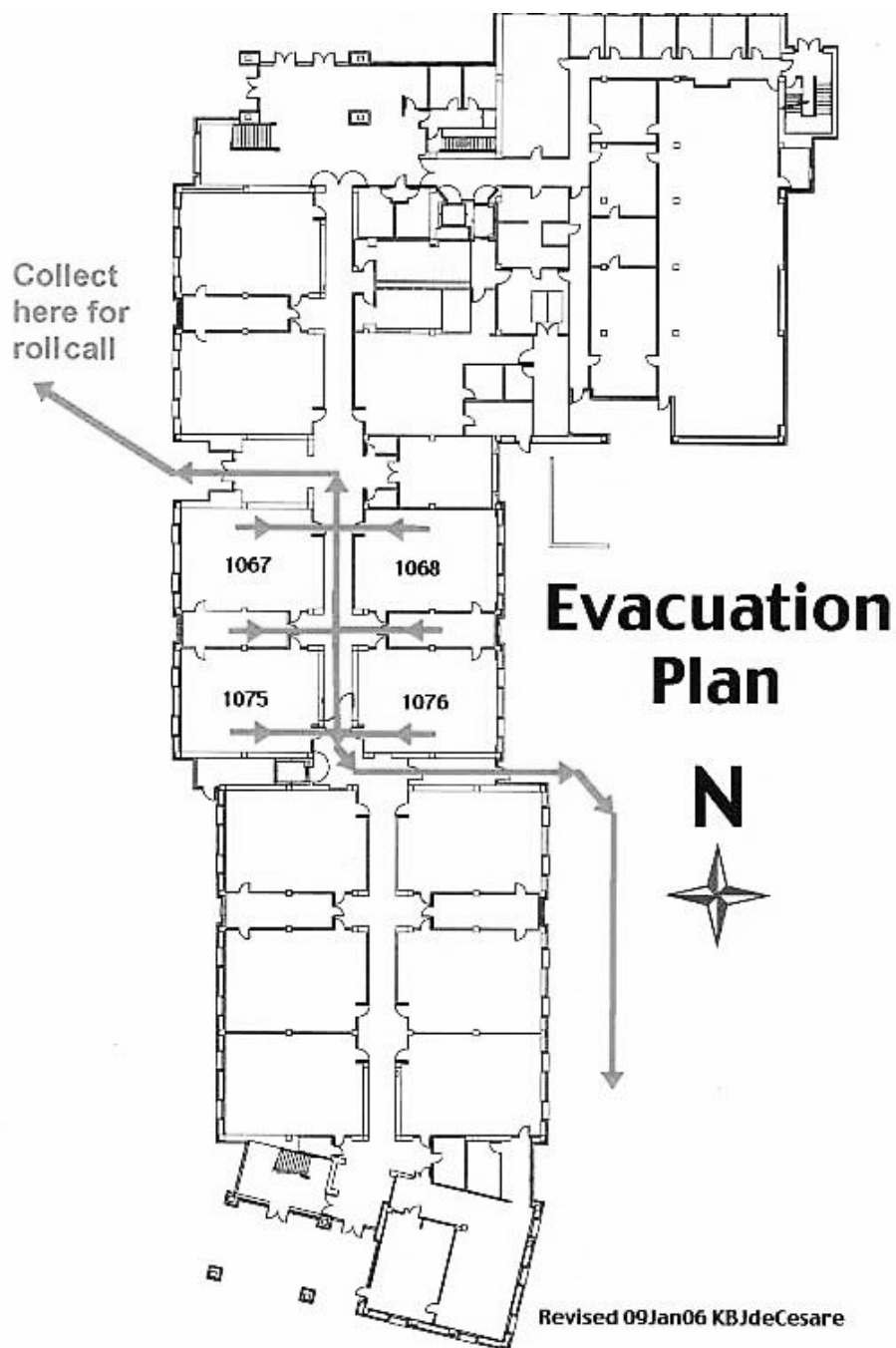
Fit

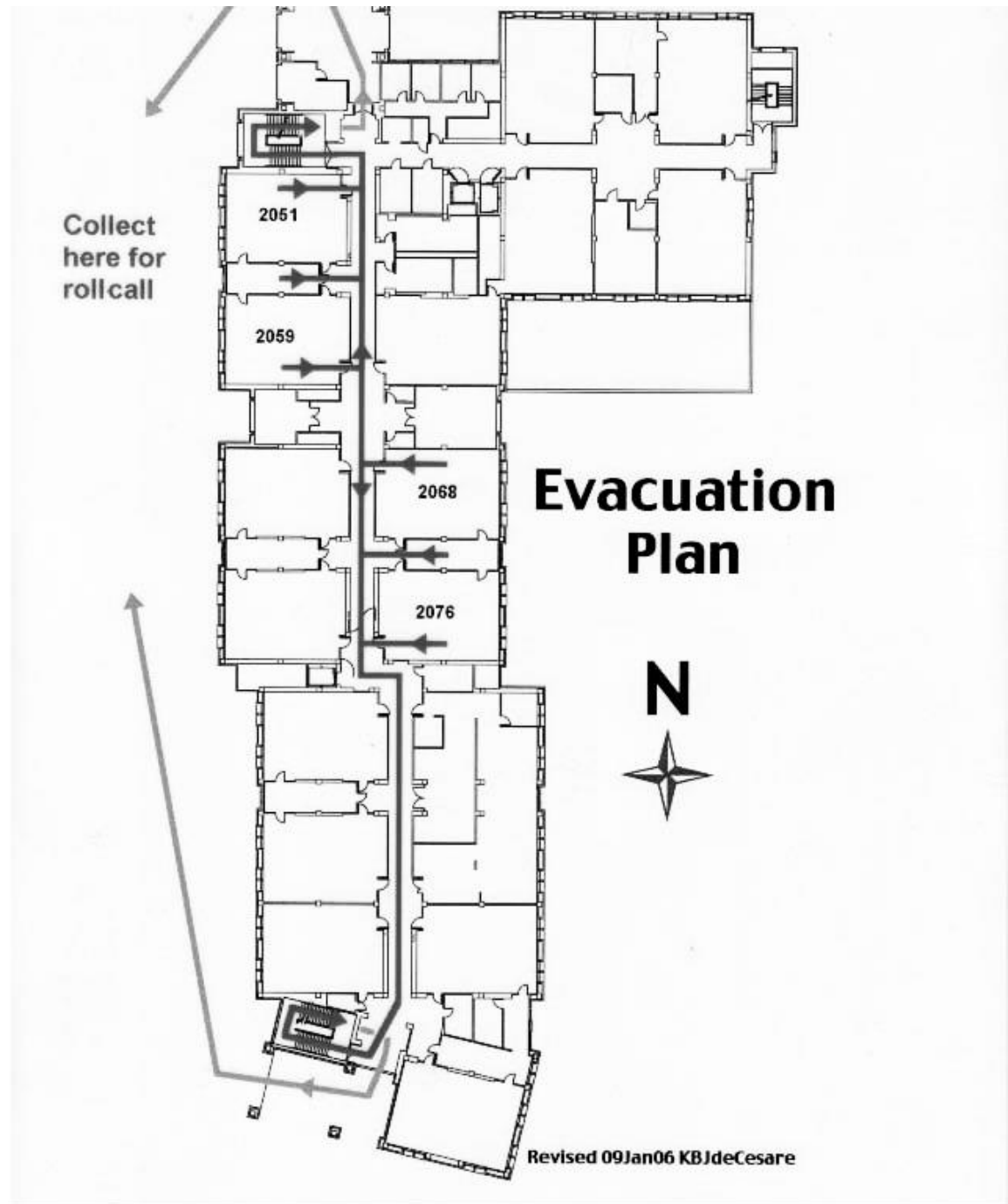
- Make sure you wear the correct sized gloves. Gloves that are too large for your hand greatly increase the likelihood of accidents.

F) Maps and Emergency Evacuation

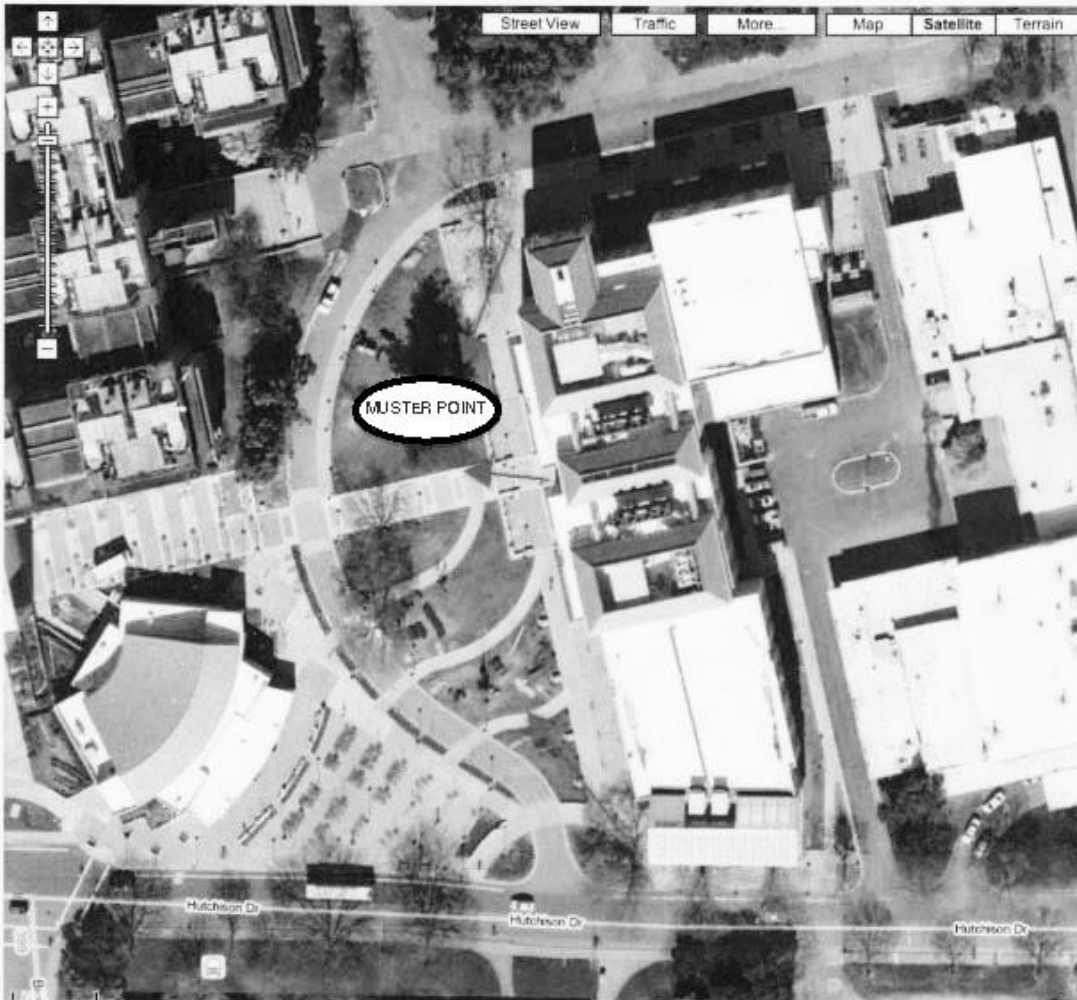


Appendix
MAPS AND EMERGENCY EVACUATION





Appendix
MAPS AND EMERGENCY EVACUATION



GENERAL PROCEDURES:

The following are some general instructions for actions to take in case of an emergency:

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.

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.

Fire Alarm

1. When fire alarm sounds, evacuate the room and follow evacuation plan to the Muster Point. The Muster Point 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.

Appendix
DISPENSARY PROCEDURES

G) Dispensary Procedures

G-1. Dispensing Policies

The following outline concisely describes the various stockroom dispensary procedures that will be used this quarter. Please read this over carefully, and discuss any questions you may have with your TA.

a. Policies at Beginning of Quarter

Goggles and Lab Coat: You are required to provide your own goggles and lab coats.

Locker Supplies There will be a two-week grace period for filling out dispensing room slips when checking out supplies from the dispensary for your locker. 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, you must bring the broken item or a representative portion thereof to the dispensary and fill out a dispensing slip for a replacement. If for some reason you are not able to bring the broken item, you must fill out a dispensing room slip and have your TA sign it before you may obtain a replacement.

Equipment on Loan from the Dispensary All equipment that is on loan from the dispensary must be returned to the dispensary at the end of each laboratory period.

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.

Waste Containers Full Cation Metal Waste containers may be exchanged for empties at the Dispensary window. Call the dispensary when special 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-In Clean and quickly dry all equipment. Replace all broken or missing items by checking them out from the stockroom. 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.

G-2. Waste Labels

Cation Metal Waste: Label is WHITE and is used in all CHEM 2 courses

HAZARDOUS WASTE Chem 2 Experiment Cation Metal Waste
Chemical Waste Composition: Bismuth, Chromium, Cobalt, Copper, Lead, Manganese, Silver, Zinc,
WASTE ONLY DO NOT LEAVE BOTTLE UNCAPPED <i>—Acidic—</i>

Organic Mixture Waste: Label is YELLOW and is used only in CHEM 2B.

HAZARDOUS WASTE Chem 2B Experiment Colligative Properties
Chemical Waste Composition: Cyclohexane, Acetone, p-Dibromobeneze, p-Dichlorobenzene, Naphthalene, Diphenyl, Benzophenone,
WASTE ONLY DO NOT LEAVE BOTTLE UNCAPPED <i>--FLAMMABLE--</i>

Appendix
DISPENSARY PROCEDURES

Dithizone in Chloroform Waste: Label is BLUE and is used only in CHEM 2C

HAZARDOUS WASTE Chem 2C Experiment Qualitative Analysis
Chemical Waste Composition: Chloroform, Acetone, Dithizone,
WASTE ONLY DO NOT LEAVE BOTTLE UNCAPPED --FLAMMABLE--

G-3. Locker Inventory

Procedure for beginning of quarter

- (1) Replace broken or missing items in your locker in the first two weeks. They may be checked out from the stockroom (Room 1060). All excess equipment should be placed in the extra glassware box (red) in the lab room.
- (2) Students Are Responsible for Providing Their Own Personal Protective Equipment. Only safety goggles which have been approved by the Chemistry Department are acceptable

CHEMISTRY 2 LOCKER LIST

GLASSWARE

- 1 100 ml Beaker
- 1 150 ml Beaker
- 1 250 ml Beaker
- 1 400 ml Beaker
- 1 800 ml Beaker
- 2 50 ml Erlenmeyer Flask
- 2 125 ml Erlenmeyer Flask
- 2 250 *or* 300 ml Erlenmeyer Flask
- 2 500 ml Erlenmeyer Flask
- 1 100mm Watch Glass
- 2 Glass Stir Rod
- 10 Test Tubes (rounded end)
- 6 Centrifuge Tubes (pointed end)
- 2 Thermometer, non-mercury
- 2 25 ml Volumetric Flask
- 1 250 ml Volumetric Flask
- 1 5 ml Volumetric Pipet
- 1 10 ml Volumetric Pipet

METAL EQUIPMENT

- 1 Beaker Tongs
- 1 Crucible Tongs
- 1 Scoopula
- 1 Test Tube Clamp

PORCELAIN

- 1 Small Casserole
- 1 Large Casserole
- 1 Evaporating Dish
- 2 Crucible
- 2 Crucible Cover

PLASTIC WARE

- 1 250 ml Washing Bottle
- 1 25 ml Graduated Cylinder (may be glass)
- 1 Short Stem Funnel (may be glass)
- 2 1 L Bottle, square
- 1 Desiccator
- 1 Pipet bulb w/ Tip
- 1 Plastic Test Tube Rack

OTHER

- 1 Centrifuge Tube Brush (pointed end)
- 1 Test Tube Brush (rounded end)
- 1 Vial, Alkacid Test Paper
- 1 Sponge
- 2 Rubber Policeman
- 1 Wire Triangle, Pipe Stem Covered
- 1 Wire Gauze Square

COMMUNITY SUPPLIES

COMMUNITY LOCKERS

- 8" Extension Clamp
- Clamp Holder
- Small Support Ring
- Large Support Ring

SHELVES

- 50 mL or 25 mL Buret

AT LAB BENCH

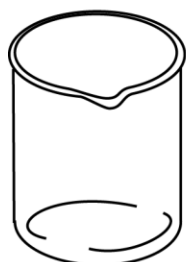
- Bunsen Burner
- w/ Silicone Rubber Tubing

Procedure for end of Quarter

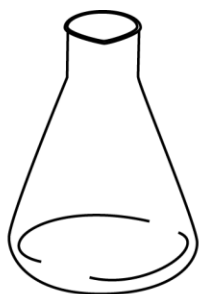
- (1) Clean and dry all equipment.
- (2) Replace all broken or missing items by checking them out from the stockroom. Return all extra equipment to the extra glassware box in lab.
- (3) Have your TA check your equipment and initial below.

Student Name _____ T.A. _____
(print) (i nitial)

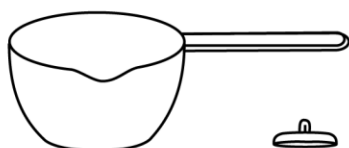
Appendix
DISPENSARY PROCEDURES



Beaker



Erlenmeyer Flask



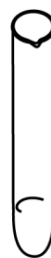
Casserole Dish



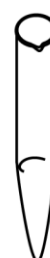
Evaporating Dish



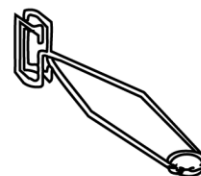
Crucible
(with Cover)



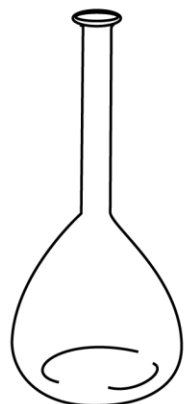
Test Tube



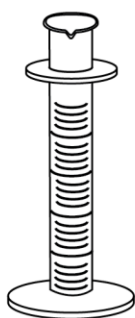
Centrifuge Tube



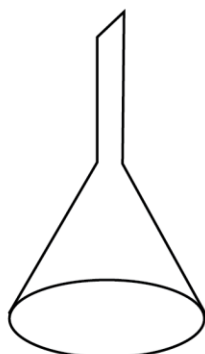
Test Tube Clamp



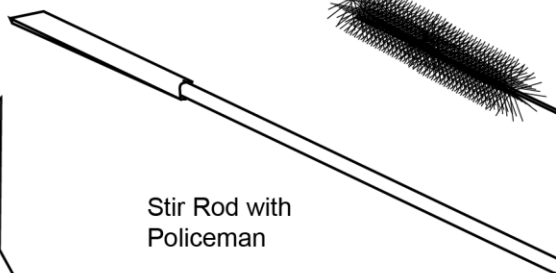
Volumetric Flask



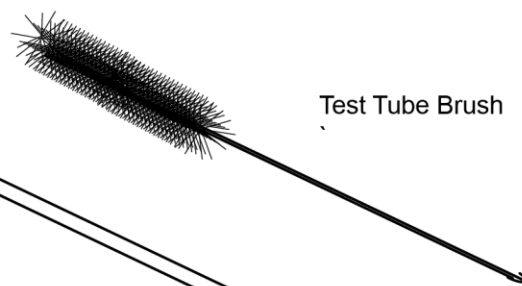
Graduated Cylinder



Funnel



Stir Rod with
Policeman



Test Tube Brush



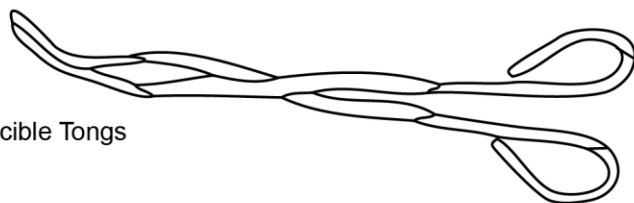
Scoopula



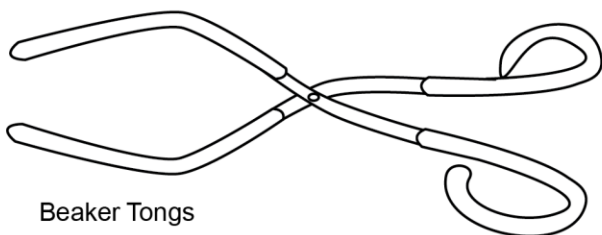
Watch Glass



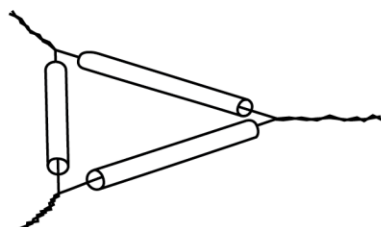
Volumetric Pipet



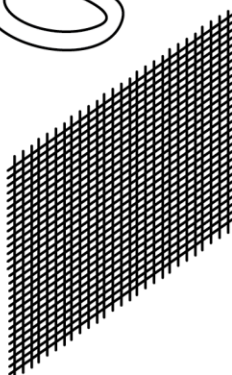
Crucible Tongs



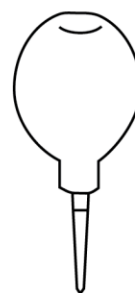
Beaker Tongs



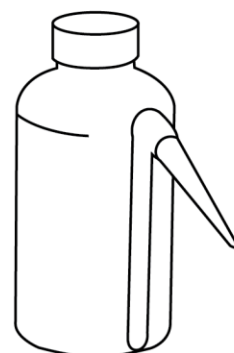
Wire Triangle



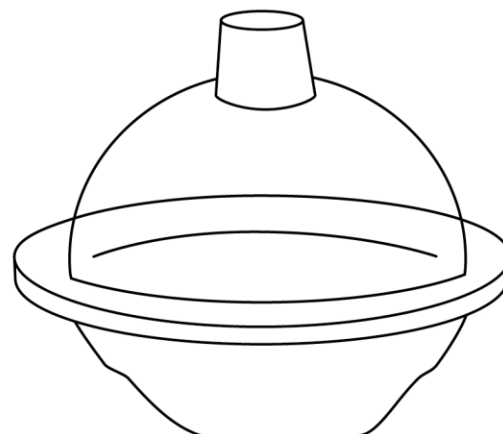
Wire Gauze



Pipet Bulb with Tip



Washing Bottle



Dessicator

18
VIII A18
VIII A

57	La	138.9
58	Ce	140.1
59	Pr	140.9
60	Nd	144.2
61	Pm	(145)
62	Sm	150.4
63	Eu	152.0
64	Gd	157.3
65	Tb	158.9
66	Dy	162.5
67	Ho	164.9
68	Er	167.3
69	Tm	168.9
70	Yb	173.0
89	Ac	(227)
90	Th	232.0
91	Pa	231.0
92	U	238.0
93	Np	(237)
94	Pu	(244)
95	Am	(243)
96	Cm	(247)
97	Bk	(247)
98	Cf	(251)
99	Es	(252)
100	Fm	(257)
101	Md	(258)
102	No	(259)