SUNY COLLEGE AT ONEONTA

General Chemistry I

Fall 2019 Lab Manual

If you are unprepared or late, your lab instructor may not allow you to complete the lab!

Before coming to lab each week do the following:

- Read the lab completely
- Complete the pre-lab in your duplicating notebook write the questions (abbreviated) and the answers
- Prepare large data tables in your duplicating notebook for the data that will need to be collected that day
- Bring goggles and wear appropriate clothing
    - Shoes must cover the foot and toes
    - Long hair must be tied back
    - No loose sleeves or scarves
    - Shorts are discouraged and must come to the knees
    - Shirts must have sleeves (no tank tops)
    - Lab coats are recommended but aprons will be provided

Revised August 2019 VL DuBois

INSIDE FRONT COVER

Fall 2019 CHEM-111 Schedule

Week starting:	Experiment	Points
Aug 26	Introduction to the SUNY Oneonta General Chemistry Laboratory	10
Sept 2	No Labs this week – Labor Day	0
Sept 9	Accuracy and Precision	6
Sept 16	Percent Yield - Synthesis of Alum	6
Sept 23	Reaction Stoichiometry – Determining a Limiting Reactant	6
Sept 30	Solution Stoichiometry – Determining the Molar Mass of an Unknown Acid	10
Oct 7	Net Ionic Equations	6
Oct 14	No labs this week – Columbus Day	0
Oct 21	Thermochemistry: Enthalpy of Formation and Dissolution	6
Oct 28	Spectrophotometric Analysis of Copper	10
Nov 4	EXAM - Analysis of a Copper Complex	20
Nov 11	Written Lab Exam and Analysis of a Copper Complex Week 2	20
Nov 18	Lewis Structures and Molecular Shapes and Check-out	6
Nov 25	No labs this week - Thanksgiving	0
Dec 2	Back-up week	
		Total = 106 pts

Solubility Rules

SOLUBLE COMPOUNDS	EXCEPTIONS
Almost all salts of Na^+, K^+, NH_4^+	
Salts of nitrate, NO_3^- chlorate, ClO_3^- perchlorate, ClO_4^- acetate, $CH_3CO_2^-$	
Almost all salts of Cl^-, Br^-, I^-	Halides of Ag^+, Hg_2^{2+}, Pb^{2+}
Compounds containing F^-	Fluorides of Mg^{2+}, Ca^{2+}, Sr^{2+}, Ba^{2+}, Pb^{2+}
Salts of sulfate, SO_4^{2-}	Sulfates of Ca^{2+}, Sr^{2+}, Ba^{2+}, Pb^{2+}, Ag^+

INSOLUBLE COMPOUNDS	EXCEPTIONS
Most salts of carbonate, CO_3^{2-} phosphate, PO_4^{3-} oxalate, $C_2O_4^{2-}$ chromate, CrO_4^{2-}	Salts of NH_4^+ and the alkali metal cations Na^+, K^+ are exceptions for all of these
Most metal sulfides, S^{2-}	BaS is soluble
Most metal hydroxides OH^- and oxides O^{2-}	$Ba(OH)_2$ is soluble

all acids are soluble

Strong Acids	Strong Bases
HCl	LiOH
HBr	NaOH
HI	KOH
HNO_3	$Ca(OH)_{2\,(s)}$
$HClO_4$	$Ba(OH)_{2\,(s)}$
H_2SO_4	

Weak Acids	Weak Bases
CH_3COOH	CH_3COO^-
NH_4^+	NH_3
H_2CO_3	CO_3^{2-}
$H_2C_2O_4$	$C_2O_4^{2-}$
H_2SO_3	SO_3^{2-}
H_2S	S^{2-}
H_3PO_4	PO_4^{3-}
HCN	CN^-
HF	F^-
NHO_2	NO_2^-
HClO	ClO^-

Gas Forming Reactions:

$2\,H^+ + CO_3^{2-} \rightarrow H_2O(l) + CO_2(g)$

$2\,H^+ + S^{2-} \rightarrow H_2S(g)$

$2\,H^+ + SO_3^{2-} \rightarrow H_2O(l) + SO_2(g)$

$2\,H^+ + M \rightarrow M^{2+} + H_2(g)$

M = a metal atom

Strong Electrolytes:
Soluble ionic compounds
Strong acids and strong bases

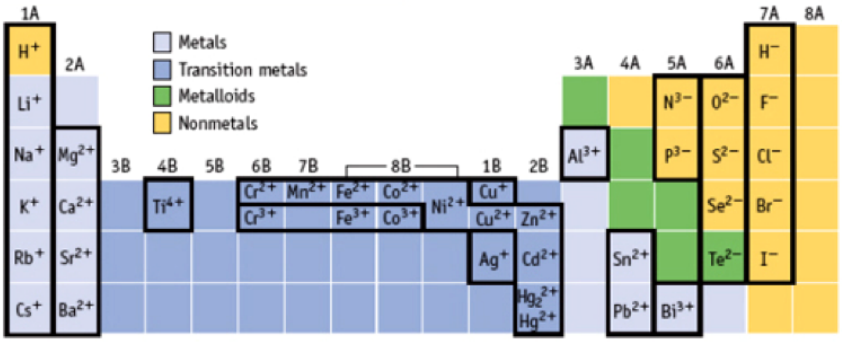


Table 3.1 Formulas and Names of Some Common Polyatomic Ions

Formula	Name	Formula	Name
CATION: Positive Ion			
NH_4^+	ammonium ion		
ANIONS: Negative Ions			
Based on a Group 4A element		**Based on a Group 7A element**	
CN^-	cyanide ion	ClO^-	hypochlorite ion
$CH_3CO_2^-$	acetate ion	ClO_2^-	chlorite ion
CO_3^{2-}	carbonate ion	ClO_3^-	chlorate ion
HCO_3^-	hydrogen carbonate ion (or bicarbonate ion)	ClO_4^-	perchlorate ion
Based on a Group 5A element		**Based on a transition metal**	
NO_2^-	nitrite ion	CrO_4^{2-}	chromate ion
NO_3^-	nitrate ion	$Cr_2O_7^{2-}$	dichromate ion
PO_4^{3-}	phosphate ion	MnO_4^-	permanganate ion
HPO_4^{2-}	hydrogen phosphate ion		
$H_2PO_4^-$	dihydrogen phosphate ion		
Based on a Group 6A element			
OH^-	hydroxide ion		
SO_3^{2-}	sulfite ion		
SO_4^{2-}	sulfate ion		
HSO_4^-	hydrogen sulfate ion (or bisulfate ion)		

Determining Net Ionic Equations

1. Write out all reactants as they exist in solution
2. Identify acids and bases
   - 2a. If both an acid and a base are present, an acid-base reaction occurs
   - 2b. Be sure to look for hidden bases that are anions in other ionic compounds, such as CO_3^{2-} in $CaCO_3$.
3. Look for ions that will form an insoluble compound. If so, they form a precipitate.
4. Look for one of the known gas-forming reactions.
5. Write out products as they exist in solution.
6. Cancel spectator ions. Note: ions that are "always soluble" will be spectator ions in acid-base or precipitation reactions.

Pre-Laboratory Assignment for: Introduction to the SUNY Oneonta General Chemistry Laboratory

No pre-lab assignment due for first week of lab. Every other lab will require a pre-lab assignment due when you enter lab.

If possible, bring a lap-top to the first lab.

Introduction to the SUNY Oneonta General Chemistry Laboratory

Objective:

This laboratory exercise is intended to familiarize you with the general chemistry laboratory. The following will be covered:

- the physical layout of the lab
- evacuation procedures
- the location of the chemicals and materials you will be using
- the procedures you will be expected to follow to complete the labs correctly and safely
- waste disposal procedures
- the correct use of balances
- graphing practice

Hazards:

- Wash and dry your bench top before use
- Wash your hands often during and before leaving the lab at any time

Procedures:

Department of Chemistry and Biochemistry
Policy on Course Attendance, Performance, Participation and Behavior

1. Students are expected to attend all scheduled course sessions and should be prepared by reading in advance any relevant material assigned or provided. Participation (defined by interacting with the instructor, working problems at the board, individually or in groups, using personal response "Clicker" systems and other mechanisms defined in the syllabus) is expected.
2. Students are reminded that instructors are not required to accept assignments submitted late, except in instances allowed according to College policies. College Policies as defined in the Student Code of Conduct apply to lecture, recitation and laboratory portions of all courses.
3. Laboratories are an integral part of education in chemistry courses. As a result, participation in all laboratories scheduled for a course is expected. Unless alternate activities are scheduled, students can expect that their laboratory section will meet each week, and failure to attend laboratories may lead to failure in the course.
4. The minimum acceptable grade for a chemistry course prerequisite is a C-. For example, a student with a D+ in General Chemistry I may not enroll in General Chemistry II. This standard applies to all Chemistry prerequisites for all Chemistry courses.
5. **The laboratory for a course must be passed**, normally by earning 60% of the available score or points for the laboratory, in order to pass the course. Exceptions may be noted in syllabus.
6. Students are expected to bring to laboratory the laboratory manual (or printout of the experiment), a laboratory notebook (if required), a calculator, ruler or other materials as specified by the instructor or in the syllabus.
7. Students are not allowed to work in the laboratory without direct faculty supervision.
8. Unless announced in advance, **SAFETY GOGGLES (WHICH PROVIDE A COMPLETE SEAL AROUND THE EYES AND ARE EQUIPPED WITH INDIRECT VENTS) ARE REQUIRED TO BE WORN AT ALL TIMES IN THE LABORATORY. STUDENTS ARE REQUIRED TO PROVIDE THEIR OWN SAFETY GOGGLES.**
9. Open-toed shoes (e.g. sandals, "Birkenstocks", flip-flops, etc), unrestrained long hair, excessively loose clothing and other items, which may be easily ignited or snag on apparatus are not allowed.
10. Food, drink, candy, cosmetics, tobacco products, etc. are not allowed in the laboratory.
11. Students are expected to be attentive to the material and any experiments and apparatus in the laboratory. The following must be turned off and stored away from the laboratory bench while in laboratories: Portable music players (e.g. iPods, MP3 players and the like); Cellular telephones, pagers, text messaging devices and the like; Other portable electronic devices as defined by the laboratory instructor.
12. Horseplay, practical jokes, "goofing around" or interfering with other students' work is not allowed in the laboratory.
13. Students should not expect to be able to makeup missed laboratory sessions or experiments. If a makeup session is possible, it will be at the discretion of the laboratory instructor and will normally be during the same week as the missed laboratory section.
14. Students will not be permitted to work in any laboratory section other than that they are registered for unless they have the **written approval** of both their regular instructor AND the instructor in the section they wish to enter.

Course instructors may modify these guidelines as necessary to meet the requirements of individual courses or chemical specialties in consultation with the Department Chairperson. Students should expect to receive a copy of these guidelines in their course syllabus or be given a copy by the course instructor (either in paper form or by electronic mail).

When Entering the Lab:

- Have pre-lab and previous week's lab report ready to hand-in when you ENTER the lab. Do not expect time to collate, trim, finish and staple.
- Put on your goggles and wash and dry your bench-top before putting anything on them
- Hang coats and place bags in the areas provided. Put your cell phone in your bag. No coats, bags or cell phones are allowed at the benches. This is for your safety and the safety of others in the lab. Bring a calculator, a duplicating lab notebook, a PEN and your lab manual to your bench. Your goggles should be on. **Put your coat, bag and phone away now if you haven't already.**

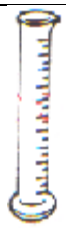
Lab Partners, Glassware Bins and Common Use Chemicals and Materials:

- Your instructor will assign partners, bench locations and glassware bins. Your instructor may switch your partner at any time throughout the semester.
- It is expected that each person come prepared to do the laboratory experiment, which includes reading the entire lab, completing the pre-lab assignment and preparing your lab notebook with data tables to record the data for that experiment.
- If you come unprepared you may be required to complete the labs alone.
- The glassware bins correspond to the location of your bench. Only the glassware listed on the laminated sheet should be returned to the bin. Common use glassware should be returned to the location from which it was obtained – CLEAN.
- All glassware must be clean when returned to the bin. Report unclean glassware to your instructor. Use tap water, soap and a brush to clean glassware. Rinse with tap water and then do a final rinse with purified water from your squeeze bottle.
- Materials you find on your bench when you enter the lab should remain on your bench when you leave. They should be clean, CAPPED, and tidy.
- Common use materials will be placed around the perimeter of the room.
    - CLEAN and RETURN all common materials to where you obtained them.
    - Keep chemicals capped/covered.
    - Clean up all spills immediately! Report spills to your instructor.

Leaving the Lab:

- You may step out to use the restroom or your phone at any time, but always wash your hands before leaving, EVERY time.
- You may remove your goggles just before leaving the lab.
- Before leaving for the day:
    - Get your NB signed by your instructor before cleaning up in case you need to repeat something.
    - Thoroughly clean your balance as described in the proper use of a laboratory balance section.

- Clean all glassware and equipment and return them as described above.
- Wash and dry your bench-top.
- Clean and tidy some other common area of the lab. Be a professional citizen.
- Wash your hands

Overview of Laboratory Glassware		
This is a guide illustrating the different types of glassware and chemistry utensils you may use this semester and their proper use. Only some are contained in your bin.		
Name	**Picture**	**Use**
Beaker		This is one of the most versatile pieces of glassware. You can use it to carry out reactions in, make solutions, make an ice or hot water bath, etc. NOT used for accurate volume measurements. Graduations are approximate.
Graduated Cylinder		This instrument is used to measure the volume of liquids.
Erlenmeyer Flask		Like the beaker, the Erlenmeyer flask can be used to carry out reactions in. It has slanted walls so it's easier to swirl your reaction without spilling.
Funnel		Use this to transfer either liquids or very fine solids into another piece of glassware.
Evaporating Dish		Use this to dry compounds quickly over a flame. For most of our labs, we dry compounds in air in our drawers instead.
Test Tube		Use test tubes to make solutions or carry out reactions.
Wire Test Tube Holder		Use this ONLY to hold test tubes. It makes it easier and safer to heat in a flame if need be.
Metal Spatula		Use this to transferred solids from the stock bottles to a weighing paper, or to mix solutions at your lab bench.
Watch Glass		Use this to carry out small reactions in open air, to cover a beaker or evaporating dish, or

		to hold crystals you synthesized and will analyze the following week in lab.
Medicine Dropper		Use this to add a solution dropwise to a reaction.
Ring Stand		Set up various clamps to this to hold your glassware in place.
Ring Clamp		Clamp this to the ring stand and place a wire gauze on top to have a level surface to place other glassware for heating.
Utility Clamp		Use this to clamp on to various glassware such as test tubes or Erlenmeyer flasks.

Laboratory Notebooks

A bound laboratory notebook with duplicating pages is required for this course and is available at the bookstore.

- All data is to be written directly into the notebook. Do not write on a paper towel, your hand etc. Bring your notebook with you to the balances or anywhere you will be taking a measurement. Your notebook is a contemporaneous diary of what you do in lab.
- **Use blue or black INK. NOT pencil or other colored ink.**
- Write firmly with the back flap under the page (and copy sheet) you are writing on.
- Do not tear out the original pages. Make copies if the duplicate sheet is illegible. DO NOT tear out pages to give to someone who has forgotten their notebook.
- Prepare your notebook BEFORE coming to lab:
    - Fill out the top information for the experiment you will be doing.
    - Complete the pre-lab questions in the notebook and be prepared to hand in the duplicates at the beginning of lab.
    - On the pages following the pre-lab, prepare your notebook to neatly record the data you expect to record.
- DO NOT try to fit the entire experiment onto one page. Write large enough to be legible and leave space to make additions and corrections.
- Cross out errors using ONE NEAT LINE, and then enter the correct data. Any erroneous notes should be legible through the cross-out line.

Pre-Laboratory Assignments

Pre-laboratory assignments are worth 10% of your laboratory grade and are to be completed in your bound laboratory notebook with duplicating pages. Write the questions and the answers. You will hand in the duplicate pages of your pre-lab when you enter the lab each week. Remove the spiral edges before handing it in. Make sure the duplicate pages are legible. If you collaborate with a classmate on the assignment, make sure you confirm all calculations independently and write in your own words. Assignments with similar wording and/or the same incorrect calculations will be subject to a zero and the instructor will follow the College's policies on academic dishonesty. Do NOT copy (or allow another to copy) a forgotten pre-lab right before lab. It is not worth it. The instructor may give a quiz at the beginning of lab to test your preparedness.

Lab Reports

You will either have a laboratory report form to complete or a formal typed lab report due the week following each lab unless otherwise instructed by your professor.

Report forms:
If a report form is provided, you will not need to type a formal report, rather you will complete and **hand-in the report form at the end of the lab session** unless your instructor directs otherwise. You must complete the forms neatly and in complete sentences if a "fill in the blank" spot is not provided. Do not submit the same wording as anyone else! Your report should NOT be the same (or virtually the same) as another student or you will be reported for plagiarism. Your efforts in lab and the results in your report form are worth 5, 3 or 0 points. Along with your pre-lab this is a total of 6 points for the week.

Typed lab reports:
This section explains how you should prepare TYPED lab reports. These labs will have a rubric table provided which you will remove from the manual and attach to the back of your report. This format should be used unless you are told otherwise. Write in the 3rd person (do not use "I weighed 1.023 g"…", instead write "1.023 g was weighed…"). **Use subscripts for formulas and superscripts for ion charges.** This is important in chemistry! Proofread your own report and consider having another student also proofread it. Fix formatting so it looks professional before you hand it in. **Tables and graphs should all be on one page, not half on the bottom of one and half on the top of the next**. Title plots and label the x- and y-axes of all graphs. See the back of this manual for a sample lab report.
Typed labs are worth 9 points for a total of 10 points with the pre-lab.

Cover sheet - your full name, your partner's full name, the day and time your lab meets, your instructor's name, the full title of the lab and the date the lab was performed.

Purpose - The purpose or objective of the experiment is rarely more than two sentences. You need to explain what you are intended to learn from completing the experiment. What are the end goals of the experiment and how did you get there (what technique)?

Procedure - You need not reproduce the procedure for the experiment unless you needed to develop it yourself; just cite it from the lab handout and note any changes. For example, "The procedure for this experiment is found in General Chemistry I laboratory Manual, Pages 56 – 59. Instead of using 100 mL of water, 50 mL was used." If there are changes to the lab during the course of the lab session, write it in your notebook so you don't forget.

Observations and Data – This section should include all the raw data you took during lab. Raw data includes measurements and observations. Anything that is important for completing calculations should be shown in the "raw data" format in this section. Measurements should be put in table format. When recording

data do not round, enter all the decimal places indicated unless otherwise specified. Numbers less than 1 should include an initial zero: "0.128 g", not ".128 g". **Observations must be recorded for each experiment.** Observations are what you sensed (saw, heard, smelled…) during the experiment, initial conditions, all the changes that were seen, the final conditions and where you believe errors could have occurred. Including:

1. the color, appearance, and physical state of reactants and products
2. temperature changes
3. gas production (bubbles)
4. precipitate formation (solid appears)
5. solids dissolving (solid disappears)
6. color changes of solutions
7. solutions becoming cloudy or becoming clear

Record your observations during the experiment. DO NOT wait until the end of the experiment to write them down. You will probably have forgotten a lot of the details or may remember them incorrectly.

Calculations – You should show all of your work for any calculations you make for a lab and explain, with words, what each number represents. Some calculations are multi-step; you should include all of those steps when you type out your work. If there are multiple calculations that are the same, show at least ONE example calculation IN THE REPORT. Attach any excel plots that are required for the calculation. Plots should be titled, and axes should be labeled. **For experiments with multiple trials, NEVER average your data. Instead, calculate each trial separately and average the results.**

Results – Summarize your calculated results in table format.

Conclusions – Report the RESULTS of the lab! If the purpose was to determine the molar mass of a substance, your conclusion MUST include what you determined the molar mass to be! Reflect back on your purpose of the lab. Explain what you can conclude about the purpose based on the observations, data and calculations. Include % yield or % difference where appropriate. **Always include sources of loss and error and explain how the error could have affected your results.** Errors are not necessarily things you do incorrectly but can instead be the result of assumptions made or simply due to aspects of the chemistry involved. This section should be at least a few sentences in length. **The conclusion is NOT a list of what procedures you learned to do or what you liked about the lab.**

Questions – If the lab had additional questions that followed the procedure, type out the questions, number/label them as the lab does, and answer the questions.

Notebook Page Duplicates – If your instructor hasn't already collected them, attach the duplicate copies (yellow) of your notebook pages to the back of your typed report or report form. If they are illegible, make photocopies of the originals and attach. Remove the spiral paper edging.

Grading and other Policies

The maximum grade for laboratory is 106 points. No labs are "dropped". This means you have the opportunity to earn the 6 points, or 6%, as "extra-credit" if you do not need to miss any labs. This extra 6 points serves as a buffer if you need to miss a lab for any reason (excused or not). If you have an excused absence, your instructor can give you a link to a video showing a lab being performed which you can use to make-up for the excused absence. You would need to write a lab report for the experiment in the video. If you have more than one excused absence in a semester your instructor will calculate a percentage based on the labs you attended. Proper documentation will be required to excuse a lab and it is at your instructor's discretion if you are exempt from it.
There will be two 20-point exams. One is similar to a hands-on "lab practical" (Analysis of a copper complex) and the other is a written exam. The written exam will test you on safety procedures, concepts learned in lab, equipment, glassware, techniques, and the calculations similar to those required in the labs you performed. NOTE: **You will be accountable for the content of ALL labs regardless of if you attended them or not. Be sure to study all of the labs and see your instructor with questions.** You NEED to make arrangements to ensure that you are able to attend these 2 exams during the weeks they are given. See your instructor during the first few weeks of lab if you have a conflict and make arrangements.

Pre-laboratory assignments are worth 1 point and may not be turned in late. 6 point labs must be handed in at the end of the lab period unless otherwise indicated by your instructor. If your instructor agrees to accept a late typed laboratory report, it must be turned in no later than the following Monday regardless of when your lab normally meets. There will be a 2-point penalty for late lab reports.

Be PUNCTUAL!! During the first part of the class, instructors will be teaching concepts that may not be covered in lecture, going over critical laboratory procedures and most importantly the safety hazards for the day. If you are late you may not be allowed to participate in lab that day!

Safety:

You are expected to look up the Safety Data Sheet (SDS) for each of the chemicals used prior to each lab. The SDS's can be found on the college web site. Your instructor will provide the location and proper use of the safety equipment in the room. **You are responsible for knowing where to find and how to properly use the following**. If you are unsure or did not understand your instructor's explanation, ask for clarification from your instructor or any chemistry professor.

- Exits and evacuation locations
  - Stay at evacuation location – we will take attendance.
  - Short term evacuation - nearest safe exit away from the building and stay together.
  - Long term evacuation – your instructor will lead you to Chase gymnasium.
- Eye wash/safety shower
- Fire extinguishers
- Hazardous waste disposal
  - Follow specific directions given for each lab
  - Nothing down the drain unless directed by your instructor
  - NO metals in the garbage
  - NO nitric acid with organic solvents
  - Close hazardous waste container after use
- Study the following "Do's and Don'ts" list as well as the American Chemical Society's safety brochure before coming to lab and refer to them and the other information in this lab to answer the questions in the lab report form.
- If you are injured, no matter how slight, inform your instructor immediately. If you are burned, tell your instructor and hold the burn under cold running water. Follow your instructor's directions.

Chemistry Lab Do's and Don'ts

The chemistry laboratory must be a safe place in which to work and learn about chemistry. Most of these statements involve just using common sense.

1. Wear chemical splash goggles at all times while you are in the laboratory.
2. Be familiar with your lab assignment **before** you come to lab. Follow all written and verbal instructions carefully. Observe the safety alerts in the laboratory directions. If you do not understand a direction or part of a procedure, ask your instructor before proceeding.
3. When entering the lab/classroom, do not touch any equipment, chemicals, or other materials without being instructed to do so. Perform only those experiments authorized by the instructor.
4. No student may work in the laboratory without an instructor present. Work only with your lab partner(s). Do not venture to other lab stations for any reason.
5. Do not wear bulky or dangling clothing.
6. Never eat or drink in the laboratory. Don't chew on the end of a pen which was lying on the lab bench.
7. Wash acid, base, or any chemical spill off of yourself immediately with large amounts of water. Notify your instructor of the spill.
8. If chemical substances get in your eye, wash the eye out for 15 minutes. Hold your eye open with your fingers while washing it out
9. Clean up spills immediately. If you spill a very reactive substance such as an acid or base, notify the people in the area and then obtain assistance from your instructor.
10. Acid spills and base spills should be neutralized before cleaning them up.
11. If you take more of a chemical substance from a container than you need, you should not return the excess to the container. This might cause contamination of the substance remaining. Dispose of the excess as your instructor directs.
12. When weighing never place chemicals directly on the balance pan. **Never weigh a hot object**.
13. Do not directly touch any chemical with your hands. Never taste materials in the laboratory.
14. Never smell anything in the laboratory unless your instructor tells you it is safe. Do not smell a substance by putting your nose directly over the container and inhaling. Instead, waft the vapors toward your nose by gently fanning the vapors toward yourself.
15. If you burn yourself on a hot object, immediately hold the burned area under cold water for several minutes. Inform your instructor.
16. Observe good housekeeping practices. Work areas should be kept clean and tidy at all times. Only lab notebooks or lab handouts should be out on the table while performing an experiment. Books and book bags should not be on the lab table. Passageways need to be clear at all times.
17. Always replace lids or caps on bottles and jars.
18. Always add acid to water and stir the solution while adding the acid. Never add water to an acid.
19. Report all accidents to your instructor.
20. Absolutely no running, practical jokes, or horseplay is allowed in the laboratory.
21. Thoroughly clean your laboratory work space at the end of the laboratory session. Make sure that all equipment is clean, and returned to its original place.

DISCLAIMER

This guide contains information and guidelines that are believed to be reliable regarding the safe use and handling of chemicals in laboratories and student classrooms. The American Chemical Society (ACS), however, does not purport, in this guide or in any other publication, to specify minimum safety or legal standards or to address all of the compliance requirements, risks, or safety problems associated with the handling of hazardous chemicals, their use, or the methods prescribed for using them in laboratories or classrooms. This guide is intended to serve only as a beginning point for information and should not be construed as containing all the necessary compliance, safety, or warning information, nor should it be construed as representing the policy of ACS.

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Doing things SAFELY is not just the right way to work– it's the only way!

SAFETY FOR INTRODUCTORY CHEMISTRY STUDENTS

The chemistry laboratory includes hazards and risks! Scientists understand the risks involved in the laboratory and have established a set of laboratory safety practices. This guide summarizes the safety rules that scientists follow in the laboratory.

PERSONAL PROTECTION

- Wear eye protection (chemical-splash-proof safety goggles)! This applies at all times to all persons in the laboratory—even guests. Contact lenses worn with goggles are acceptable, but safety glasses and prescription safety glasses without goggles do not provide adequate protection. Increase the degree of protection (use face shields, laboratory hoods, etc.) when the hazards increase.
- Take care not to ingest anything in the laboratory! Food, gum, beverages, and tobacco products are never allowed in the laboratory.
- Do not apply cosmetics in the laboratory.
- Never pipet by mouth!
- Clothing should protect you from accidental spills and splashes. Wear clothing you can remove easily in case of accident. Clothes should cover the body from the neck to at least the knees.
- Tie back long hair and remove jewelry before entering the laboratory.
- Wear practical shoes. Shoes with high heels, open toes, or made of woven materials are not allowed in the laboratory. Sandals are not appropriate!
- Nonflammable, nonporous lab aprons afford excellent protection. Always remove lab aprons or coats before leaving the laboratory.
- You will often need to wear gloves to avoid skin contact with hazardous materials.
- Always, even after wearing gloves, wash your hands with soap and water before leaving the lab!

LABORATORY PROTOCOL

- Your responsibility to prevent accidents in the laboratory extends to others in addition to yourself. The laboratory experience should be enjoyable but is serious; it is not a time for play. Be aware of what is happening around you and report any questionable behavior.
- Always plan laboratory work before executing it. Providing for safety and avoiding potential accidents are important elements of the plan. You should understand the hazards associated with the chemicals involved before you start the experiment. If you are unsure about the hazards and the protection that you need, Material Safety Data Sheets (MSDSs), which provide detailed information, are available from your instructor, or at http://msdssearch.com/.
- Use a safety shield when working with highly reactive chemicals and mixtures.
- Flames are never allowed when flammable gases or liquids are in use.
- Always alert others before lighting a flame!
- Know where to find and how to use all emergency equipment (such as fire extinguishers, eye washes, and safety showers) in the laboratory.
- Always assemble laboratory apparatus away from the edge of the laboratory bench.
- Always check your glassware and discard any with chips, breaks, or obvious flaws.
- When using a laboratory hood, set the equipment and chemicals back at least 15 centimeters from the hood door.
- Be certain that you understand the proper use and operation of all laboratory equipment.
- Never work in the laboratory alone.
- Never leave experiments unattended unless you take special precautions to avoid accidents and you notify the responsible individuals.
- Use laboratory hoods for all operations in which toxic, corrosive, irritating, or flammable chemicals are involved. Be certain that the hood is operating properly prior to execution of your work!
- Never put your face inside the laboratory hood.
- Never perform unauthorized experiments or deviate from the experimental plan. Report unauthorized experiments to the instructor.

HOUSEKEEPING

- You are responsible for ensuring that a clean workspace is maintained both in your own working area and in the common working areas. The laboratory environment should be at least as clean and orderly when you finish your work as when you began.
- Place broken glassware in the proper receptacles. Do not allow it to accumulate on the bench top.
- Keep laboratory benches free of spilled chemicals. Clean up spills immediately as directed by your instructor.
- Avoid physical hazards by keeping drawers and cabinets closed.
- Prevent tripping and contamination hazards—never place materials on the floor.
- Always clean glassware before returning it to storage. Do not allow dirty glassware to accumulate.
- Follow your instructor's directions for disposal of chemicals. If you don't know, ask! Improper disposal results in possible personal hazard or environmental contamination.
- Contaminated wastepaper must be handled separately from normal wastepaper.

This guide briefly reviews the major principles that guide scientists in performing their laboratory work safely. The American Chemical Society publication *Safety in Academic Chemistry Laboratories* (http://chemistry.org/committees/ccs) provides more detailed information both about these topics and about managing the hazards of specialized operations.

Copy the following tables into your duplicating notebook.

Data table for balance/hot-plate procedures:

DIRECT ADDITION METHOD:

Identity of material	
Mass of sample (g)	

WEIGHING BY DIFFERENCE METHOD:

Identity of material	
Mass of boat AND sample (g)	
Mass of "dirty" boat (g)	
Mass of sample (g)	

Data table for pipetting:

Trial 1 (g)	
Trial 2 (g)	
Trial 3 (g)	
Trial 4 (g)	
Trial 5 (g)	
Range (largest-smallest)	

Read the following:

Follow the procedures outlined below. Record raw data in your duplicating notebook and complete the following Laboratory Report Form. Record all information requested neatly. Complete as much as you can before the end of the lab. Your instructor can assist you with the graphing practice exercise if you brought a lap-top. Before leaving, obtain a copy of a laboratory accident article from your instructor. You will need the article to complete the final section of the report. Next week's pre-lab and this completed report are due when you arrive next week. Have it removed from the manual and stapled before coming to lab.

Procedure:

Proper use of a hot-plate stirrer:

Each lab bench has a hot-plate stirrer which will be used to heat liquids and/or stir solutions. It is your responsibility to ensure that your hot-plate remains clean and in good working order. Hot-plates are fragile and expensive!

Safety:

- The hot-plate surface appears the same whether it is hot or cold. Look at the indicator light to see if the surface is still hot.
- Never move a hot-plate when it is hot.
- Be sure to keep all near-by materials away from the hot-plate including its own electrical cord!

Before use:

- Make sure nothing is touching the surface of the hot-plate.
- Ensure the hot-plate is on a level surface.

Using the hot-plate:

- With the vessel to be warmed on the center of the plate, use the knob to turn-on the heat.
- Do not use the highest setting, start at a low setting and increase as needed.

Using the stirrer:

- With the vessel to be stirred on the center of the plate, add a magnetic stir-bar into the vessel.
- Use the knob to start the bar spinning.
- Do not use the highest setting, start at a low setting and increase as needed.
- Move your vessel if needed to place the stir-bar in the center of your vessel.
- If the stir-bar loses contact with the magnet in the plate, it will start to jump around. If this happens, immediately turn the stir knob all the way back to off and let the bar settle back to the plate magnet. Re-start the spinning, but this time do not turn it up as high.

Proper use of a laboratory balance:

Each lab bench has a laboratory balance, which will be used to obtain masses of chemicals and materials used throughout the semester. It is your responsibility to ensure that your balance remains clean and in good working order. Balances are fragile and expensive! Do NOT lift, bump, or drop them.

Before use:

- Make sure the balance is on a firm level surface. Level the balance if a leveling bubble is on your balance.
- Power the balance on.
- Ensure the balance is clean.
- Press the Tare/Zero button and ensure a stable zero reading.

After each use:

- With the dry brush provided, clean above and below the pan as shown by your instructor.
- If needed, remove the pan to wash with soap and water. Dry thoroughly before replacing.
- Wipe bench-top around balance with wet sponge or paper towel and dry.
- Cap the material you were weighing!

DIRECT ADDITION METHOD (Weighing directly):

- Have your lab notebook (NB) with you at the balance. Do not try to remember the number or read it to your partner to record.
- Place vessel you will be weighing into on the center of the balance
    - Weigh-boat today, but in the future it could be a beaker, beaker holding a test-tube etc.
- Press the Tare/Zero button and wait for the reading to stabilize on zero. Press again if needed.
    - Wind currents can cause fluctuations. Use the guard attached to the balance or otherwise protect the pan from air currents and re-tare.
- Carefully add about 2 grams of the material provided into the vessel.
    - You rarely need to measure an exact amount. If the procedure says to obtain about 2 g, you should measure an amount that is close, but NOT exactly 2.000 g!! Adding and removing material to force an exact mass is not good laboratory practice. Instead, measure an amount between 1.9 g to 2.1 g.
    - Add in small amounts at a time. If too much is added, remove but DO NOT return the material to the original bottle. This could contaminate the entire bottle. Put it in a container labeled “waste.”
    - ALWAYS record ALL of the digits displayed on the balance directly into your laboratory notebook. DO NOT round.
- Transfer your material into a 125 mL Erlenmeyer flask.

- To make sure that all of the material is transferred, rinse the boat into the flask with 20 mL of purified water measured with a graduated cylinder.
- Add a stir-bar and place the flask on the hot-plate stirrer.
- Turn the stirrer on and stir gently until the solid is dissolved. See proper use of hot-plate/stirrer above.
- Remove the flask and show your flask and weigh-boat to your instructor.
- Dispose of the solution as directed by your instructor.
- Throw the weigh-boat in the trash. We do not re-use weigh-boats.
- Rinse the flask and the stir-bar with tap water then a final rinse with purified water.
- Repeat the above procedure but use the "weighing by difference" method.

Data table for balance/hot-plate procedures

(Record this table in your duplicating notebook under "Data")

DIRECT ADDITION METHOD:

Identity of material	
Mass of sample (g)	

WEIGHING BY DIFFERENCE METHOD:

- With nothing on the pan, press the Tare/Zero button and wait for the reading to stabilize on zero. Press again if needed.
    - Wind currents can cause fluctuations. Use the guard attached to the balance or otherwise protect the pan from air currents and re-tare.
- Place a clean weigh-boat on the center of the balance and note its empty mass.
- Carefully add about 2 grams of the material provided into the weigh-boat using the same guidelines as above so that the mass added is the amount required. For example, if you need to measure about 2 g of a solid material and your weigh boat was 1.255 g, add the solid until the reading displays between 3.0 g and 3.4 g.
    - Record the mass of the weigh-boat and the material in your NB. Always record all digits displayed.
- Transfer the material to your flask/beaker/reaction vessel. You do NOT need to ensure all of the material is transferred. Some may remain in the boat. This is the big benefit of using this method.
- Tare/zero the balance.
- Place the weigh-boat onto the balance and record its mass as "dirty weigh-boat."

- Subtract the "dirty weigh-boat" mass from the "weigh-boat and material" mass to obtain the mass of the material actually transferred to your flask/beaker etc.
    - Continuing the example above: If your material and weigh-boat was 3.288 g and the dirty weigh-boat was 1.304 g, the mass of the material would be 3.288 g – 1.304 g or 1.984 g. Notice that this is not exactly 2 grams as listed in the procedure. That is fine. You just need to be close with all of the digits recorded.

Data table for balance/hot-plate procedures

(Record this table in your duplicating notebook under "Data")

WEIGHING BY DIFFERENCE METHOD: (0.5 pts)

Identity of material	
Mass of boat AND sample (g)	
Mass of "dirty" boat (g)	
Mass of sample (g)	

Proper use of a volumetric pipette:

- Volumetric pipettes will be used throughout this course to deliver specific volumes of liquid. Follow these steps:
- Obtain a 10 mL pipette and a bulb/pump. **Safety note: Always hold the pipette by the upper stem near the top, especially when placing the bulb or pump. NEVER force the bulb/pump onto the pipette! It should go on just enough to be able to pull a vacuum and draw up the liquid. The pipette can snap and impale your hand.**
- Pour the liquid to be pipetted into a beaker or other suitable vessel that the pipette can fit into at a depth that will allow the tip to remain submerged even once the pipette has been filled. Today, obtain about 50 mL purified water in a 100 mL beaker. Tare the beaker on the balance.
- Using a pipette bulb or pump, draw a small amount of liquid into a 10 mL pipette. Working over a waste container, slowly move the pipette around to thoroughly pre-rinse the entire inner surface with the liquid and drain to waste. Today the waste water may go down the drain.
- Using a pipette bulb or pump in your non-dominant hand, draw the liquid past the line on the stem of the pipette.
- Remove the bulb/pump and quickly place your index finger (not thumb) over the top of the pipette, "catching" the liquid in the pipette above the line. If the liquid is at or below the line try again.
- Wipe the pipette tip with a clean paper towel or Kim-wipe.
- With the pipette still over the original beaker, slowly release your finger to drop the level of the liquid until the bottom of the meniscus is on the line.

- Place the pipette over the new vessel and release your finger. The liquid will drain but a small amount will remain in the tip. Leave it there, do not attempt to "blow" it out. If, however, there is a drop on the end, touch it to the inside of the vessel to capture it. Record the mass and re-tare the balance with the beaker on the pan.
- Ask your instructor for a demonstration if needed.
- Once you have practiced the technique a few times, dispense 10 mL into a 50 mL beaker 5 times and record the mass each time. EACH person must have a unique data set from their OWN pipetting.
- The range of the 5 trials should not exceed 0.1 g
- Repeat until you have 5 trials within the range.

Data table for pipetting: (1 pt if range is within 0.1g)

(Record this table in your duplicating notebook under "Data")

Trial 1 (g)	
Trial 2 (g)	
Trial 3 (g)	
Trial 4 (g)	
Trial 5 (g)	
Range (highest-lowest)	
Average	

Graphing in Excel Practice (3 pt)

Complete the graphing practice exercise below in Excel, the complete version – NOT the "on-line version" and attach the print-outs to this report form before handing in. The complete version of excel is free to download from the college website.

Using the instructions below and the on-line graphing tutorial links provided, create all graphs as described. For each, print the graph and data set from excel and attach them to your laboratory report form. Bookmark the links so you can refer to these tutorials throughout the semester when graphs are required.

For each of the following, enter the data set given into Excel, make the charts as instructed, print both the data set and the chart for each to attach to your report. No need to make the column headings colored or shaded.

PLOT #1

Scatter Plot with a trend-line:

A scatter plot will be used in this course when one variable that is controlled effects another variable. The variable being purposefully manipulated is called the independent variable and is plotted on the horizontal axis. In this case our independent variable is concentration. The measured or dependent variable is plotted on the vertical axis. Absorbance is our dependent variable and is a quantity you will learn about in this course. It is measured with a spectrophotometer which shines a single wavelength of light on a sample and measures how much light the sample absorbs.

You will be adding a "trend-line" to your data set which is the straight line that passes closest to all of the points as possible (called the line of best fit.) This line can be used to predict the relationship between these 2 variables even in between the points that were generated in the experiment! For example, you can read the absorbance of an unknown sample and predict its concentration.

Conc. (g/L)	Absorbance
69.8	0.781
73.4	0.813
76.1	0.847
78.4	0.868
79.3	0.868
60.5	0.677
68.3	0.757
82.3	0.916
80.2	0.888
63.6	0.696
83.1	0.930
76.3	0.845
74.8	0.833
63.5	0.703
87.2	0.954

The following link explains how to create a Scatter plot and control its appearance (e.g., labels, axis scales, etc.) What look like spaces in the links are actually underscores "_"

https://oneonta.service-now.com/kb_view.do?sysparm_article=KB0010767

This link may also help if you are using a Mac.

https://oneonta.service-now.com/kb_view.do?sysparm_article=KB0010675.

Using these instructions, create a graph of the above data set that has the following features:

1. Format the axis so the x starts at 55, and the y starts at 0.5. Add additional tick marks to both of the axes. (Excel will automatically select the bounds on the axes so that your data fills the graph - which can be problematic when trying to compare two graphs. This is why setting the axis scale manually is often best practice.)
2. Label the axes: y="Absorbance" and x= "Concentration in g/L" (Style guides for most scientific journals recommend using sentence case for axis titles. The recommended titles should reflect this and have the first letter capitalized.)
3. Add the title "Effect of concentration of compound A on absorbance at 650nm, (your name)"
4. Add a linear trend-line and display the equation and R-squared value on the graph

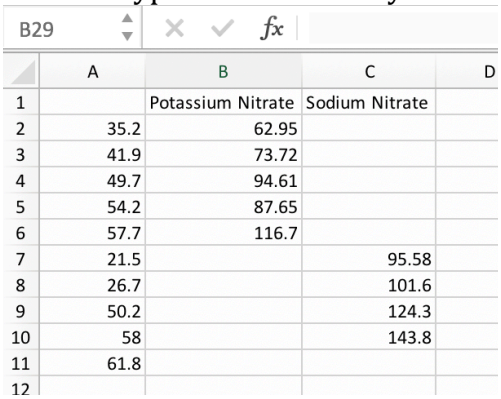
NOTE: The on-line instructions describe a "+" icon next to the chart. If you do not have a "+" icon, select the "chart design" tab at the top and the "add chart element" button in the upper left to add something to the chart. Select the "Format" tab at the top and click on the various elements in the chart. You will see the pane on the right change when you click the data, trend-line, axes etc. which will allow you to make the desired modifications.

Also, for Mac users, one can right-click by holding down the "control" key and clicking.

PLOT #2

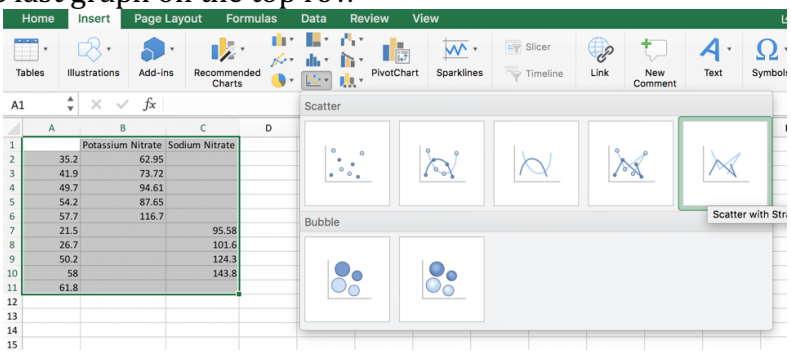
Scatter plot with 2 series of data:

1. Type the temperatures shown below in column A beginning at row 2
2. Label column B, “Potassium Nitrate” in row 1 and type in the solubility values given
3. Label column C, “Sodium Nitrate” in row 1 and type in the solubility values given

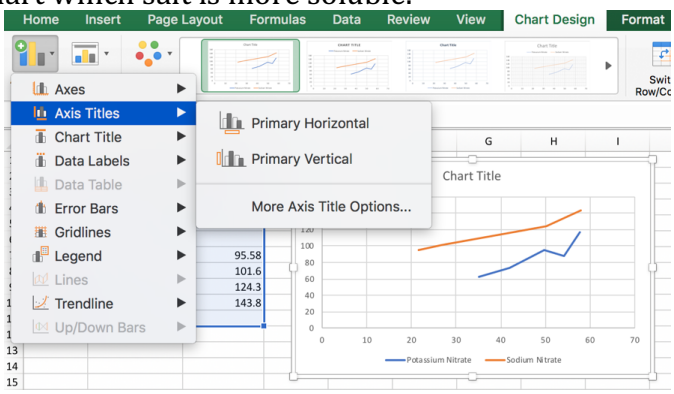


	A	B	C	D
1		Potassium Nitrate	Sodium Nitrate	
2	35.2	62.95		
3	41.9	73.72		
4	49.7	94.61		
5	54.2	87.65		
6	57.7	116.7		
7	21.5		95.58	
8	26.7		101.6	
9	50.2		124.3	
10	58		143.8	
11	61.8			
12				

4. Highlight your area containing the data and click on “insert” then the icon for scatter plot and select the last graph on the top row



5. Label the x-axis “Temperature in degrees C” and the y-axis “Solubility in g salt/100g water.” Add the title “Solubility of select salts – (your name)” To do so, click on “Add Chart Element” in the top left corner or click the “+” sign next to the graph to add a chart element. Indicate on the chart which salt is more soluble.



PLOT #3

Bar/Column graph:

A bar graph uses either horizontal bars to visually display comparisons of data in categories. Similarly, a column graph displays these comparisons in vertical bars. The categories are usually qualitative (not numerical). One axis of the chart lists the categories being compared, and the other axis represents the value of that category.

The measurement is time in seconds. Do not put the average values into excel.

Trial	Catalyst A	Catalyst B	Catalyst C
1	14	17	8
2	13	10	9
3	13	16	6
4	15	8	8
5	17	9	7
Avg.	14.4	12	7.6

Use the following link for help to create a "clustered column" graph.

https://oneonta.service-now.com/kb_view.do?sysparm_article=KB0010766

Using these instructions, create a graph of the above data set that has the following features:

1. Label the axes as appropriate
2. Add appropriate gridlines
3. Come up with a possible experiment that this data could have come from and title the graph to describe it. Be sure to include your name.
4. If a catalyst is used to speed up a reaction, indicate which catalyst is the most effective on your graph.
5. Circle the parts of the graph you used to draw this conclusion **and explain**.

PLOT #4 and #5

For both of the following sets of data:

1. Decide which type of graph to use and create the graph
2. Label axes and title as appropriate
3. Adjust the scale of the axes and add additional tick marks if needed
4. If you use a scatterplot, add a linear trend-line and display the equation and R2 value
5. Print the data set and graph
6. Draw one conclusion from the graph and write it somewhere on the graph

Data Set for Plot #4

Bagel	# Sold
Poppy seed	25
Plain	18
Everything	32
Raisin	15
Onion	29
Cheese	10

Data Set for Plot #5 (the student number should not be graphed. It is not a measurement and was assigned randomly)

Student	Hours spent studying	Test score
1	3	80
2	5	90
3	2	75
4	6	80
5	7	84
6	1	55
7	2	64
8	0.5	48
9	1	42
10	7	100
11	1.5	81
12	2.5	82
13	3.5	82
14	4	91
15	1.5	61

Introduction to SUNY Oneonta General Chemistry Laboratory Report Form

Name_____ Partner_____

Prelab (none)

Data

Data table for balance/hot-plate procedures

DIRECT ADDITION METHOD: (0.5 pts)

Identity of material	
Mass of sample (g)	

Data table for balance/hot-plate procedures

WEIGHING BY DIFFERENCE METHOD: (0.5 pts)

Identity of material	
Mass of boat AND sample (g)	
Mass of "dirty" boat (g)	
Mass of sample (g)	

Data table for pipetting: (1 pt if range is within 0.1g)

(Record this table in your duplicating notebook under "Data")

Trial 1 (g)	
Trial 2 (g)	
Trial 3 (g)	
Trial 4 (g)	
Trial 5 (g)	
Range (highest-lowest)	
Average	

Graphing in Excel Practice (3 pt)

Complete the graphing practice exercise and attach the print-outs to this report form before handing in.

Safety and Laboratory Procedure Questions: (4 pts)

Answer the following questions from the information written in this lab or given verbally from your instructor. Fill in the blanks or **use complete sentences**. **If the information is from the written lab, for credit ***copy the same wording from the lab.***

1. What are All of the things not allowed in the laboratory to reduce the possibility of **accidental ingestion** of chemicals?

2. Draw a beaker. Describe what it is used for. Describe what it is NOT used for.

3. Summarize what you do when entering the lab each week.

4. You will automatically fail the course if you earn below what grade for lab?_____

Answer the following questions from the information written in this lab or given verbally from your instructor. Fill in the blanks or **use complete sentences**. **If the information is from the written lab, for credit ***copy the same wording from the lab.***

5. Students must earn at least a _____ (letter grade) for CHEM-111 to count as a pre-requisite for CHEM-112.

6. Can you use pencil in your duplicating notebook? _____

7. Can you arrive late to lab? _____

8. What should you do if you are unsure of how to dispose of a solution?

9. Describe the location of all of the eye-wash stations and how to use them.

10. Describe the steps to wash laboratory glassware.

11. What will you do in the event of an evacuation?

12. What do you have to do before leaving the lab at any time? _____

Answer the following questions from the information written in this lab or given verbally from your instructor. Fill in the blanks or **use complete sentences**. **If the information is from the written lab, for credit ***copy the same wording from the lab.***

13. Describe **ALL** of the things you have to do before leaving at the end of the lab?

14. When making a solution of an acid, do you add acid to water or water to acid?

15. Describe what you should wear to lab.

16. Describe what you should not wear to lab.

17. What should you do if you burn yourself in the laboratory?

Laboratory Accident Article Review: (1pt)

Read the article about a laboratory accident that your lab instructor gave you. Type the following in paragraph format (about one page) and attach to this report:

a. Summarize the accident. Include the location, situation, people involved etc.
b. Describe why the accident occurred.
c. Offer suggestions as to how the accident could have been prevented.

Don't forget:

Attach the data sets and 5 plots from the graphing practice exercise as well as the summary of the lab accident article to this report form!

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Pre-Laboratory Assignment for: Accuracy and Precision

WRITE the QUESTIONS and the answers in your laboratory notebook for all pre-lab assignments. Hand in the duplicate copy when you ENTER the lab each week.

NOTE: Many words have more than one meaning. Only definitions that relate to chemistry or the way it relates to the lab will be accepted. The text glossary is a good place to start (not Google).

1. Vocabulary: (Please define or describe the following)
    a. Accuracy –
    b. Precision –
    c. Significant figures (digits) –

2. **Read the lab.** In your own words and with at least a few complete sentences:
    a. Describe the purpose of the lab. Why are we doing it?
    b. What concepts and/or calculations will we be using?

3. Draw a picture and label each type of glassware you will be using in this lab.

4. What do you do if you discover a chipped or cracked piece of glassware?

Accuracy and Precision

Objective:
This laboratory exercise is intended to determine the relative accuracy and precision of several pieces of laboratory glassware, to teach you basic laboratory techniques, and the importance of recording the maximum number of significant figures in every measurement.

Hazards:
- Wear goggles at all times
- Wash your hands before leaving the lab at any time
- Dispose of any broken or chipped glassware in the broken glass bin. Never in the garbage cans.

Laboratory Techniques:
The objective of this experiment is to prepare for future experiments by learning basic laboratory techniques important in chemistry and the importance of accuracy and precision in measurements. These include the measurement of mass, volume, temperature. In this experiment the balance will be used to determine mass. Volume will be measured using a graduated cylinder, a pipette and a burette, and temperature will be measured with a temperature probe.

MASS/VOLUME/TEMPERATURE
In this experiment you will compare the precision and accuracy of 3 pieces of volume delivering glassware: graduated cylinder, burette, and volumetric pipette

You will determine the proper number of significant figures for each measurement made with graduated scales. The last significant digit is estimated between the smallest graduations of the glassware.

It is important to report the correct number of significant figures when performing calculations with measurements.

When adding or subtracting, the answer is rounded to the least number of decimal places.
For example: 52.31 mL – 2.1 mL = 50.2 mL

When multiplying or dividing, the answer is rounded to the least number of significant figures.
For example: density = 23.344 g/67.4 mL = 0.346 g/mL

The mass of the water delivered from each will be determined and the density calculated.
The mass of a liquid varies directly with its volume, but the ratio is constant at constant temperature and pressure. The mass to volume ratio is called density and can help identify substances.

Density = mass/volume

The reproducibility of successive measurements (precision) and the agreement of the measurement with the accepted value (accuracy) of the pieces of measuring equipment will be compared.

PROCEDURE

Note: All measurements are to be recorded directly into your notebook (NB) in INK. Use ONE line to cross out and correct when needed. See the end of this lab for suggested data tables.

1. In a 250 mL beaker add approximately 150 to 200 mL of purified water and allow it to come to room temperature. Before you begin your measurements, read the temperature of the water using your temperature probe. Record the temperature (all digits, no rounding) in your notebook.
2. Tare a clean dry 50 mL beaker on a balance. Include all figures on the balance and use the same balance throughout.
3. Measure between 10 and 15 mL of water using a 100 mL graduated cylinder. Record the volume measured to the tenth of a mL and record the number of significant figures. Measure from the bottom of the meniscus estimating the last digit between the markings.
4. Transfer the water from the graduated cylinder to the (pre-tared) 50 mL beaker. Record the mass of the water. Repeat the measurement twice, re-taring the beaker in between each measurement.
5. Empty and re-tare the beaker.
6. Pipetting practice: Use your 250 mL beaker with purified water to practice pipetting until you feel comfortable with your technique. Use your finger to control the dispensing instead of your thumb or the pipette pump. Measure exactly 10.00 mL of water using the 10 mL volumetric pipette. The pipette will dispense 10.00 mL when the meniscus is set to the line on the upper thin portion of the pipette and allowed to drain. Do not force the remaining liquid from the tip. Allow the water to drain into the dry beaker. Recall that there is only one volume that can be measured using a volumetric pipette. Do not read the volume of water using the markings on the 50 mL beaker or a graduated cylinder. Record the mass of the water. Repeat the measurement twice, re-taring the beaker in between each measurement.
7. Fill a burette to about half with room temperature purified water. Deliver between 10 and 12 mL of water from the burette into an empty tared 50 mL beaker. Record the initial and final volumes measured from the burette to two decimal places. Measure from the bottom of the meniscus estimating the last digit between the markings. Record the mass of the water. Repeat the measurement twice, re-taring the beaker in between each measurement.
8. Determine the density range (Range = highest density value – lowest density value) and average density of the water for each set of measurements.

INTERPRETING YOUR DATA

Precision is an indication of ability to reproduce a measurement, so determining the range of the densities (difference between the highest and lowest values) allows you to compare the precision of the 3 pieces of measuring glassware. The smaller the range, the more precise the measurement technique was. In the conclusion rank the glassware in order of least to most precise.

The accuracy indicates how closely the measurement agrees with the accepted or theoretical value. Use your recorded temperature and the theoretical density for water. The theoretical density is obtained from the table below.

The table below provides theoretical densities of water **in g/mL** at different temperatures. Whole degrees are down the first column and tenths are across the top. To find the density of water at 16.3 °C, search down the first column to 16 then across to 0.3 to find the theoretical density of 0.998893 g/L.

	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
0	0.999841	0.999847	0.999854	0.999860	0.999866	0.999872	0.999878	0.999884	0.999889	0.999895
1	0.999900	0.999905	0.999909	0.999914	0.999918	0.999923	0.999927	0.999930	0.999934	0.999938
2	0.999941	0.999944	0.999947	0.999950	0.999953	0.999955	0.999958	0.999960	0.999962	0.999964
3	0.999965	0.999967	0.999968	0.999969	0.999970	0.999971	0.999972	0.999972	0.999973	0.999973
4	0.999973	0.999973	0.999973	0.999972	0.999972	0.999972	0.999970	0.999969	0.999968	0.999966
5	0.999965	0.999963	0.999961	0.999959	0.999957	0.999955	0.999952	0.999950	0.999947	0.999944
6	0.999941	0.999938	0.999935	0.999931	0.999927	0.999924	0.999920	0.999916	0.999911	0.999907
7	0.999902	0.999898	0.999893	0.999888	0.999883	0.999877	0.999872	0.999866	0.999861	0.999855
8	0.999849	0.999843	0.999837	0.999830	0.999824	0.999817	0.999810	0.999803	0.999796	0.999789
9	0.999781	0.999774	0.999766	0.999758	0.999751	0.999742	0.999734	0.999726	0.999717	0.999709
10	0.999700	0.999691	0.999682	0.999673	0.999664	0.999654	0.999645	0.999635	0.999625	0.999615
11	0.999605	0.999595	0.999585	0.999574	0.999564	0.999553	0.999542	0.999531	0.999520	0.999509
12	0.999498	0.999486	0.999475	0.999463	0.999451	0.999439	0.999427	0.999415	0.999402	0.999390
13	0.999377	0.999364	0.999352	0.999339	0.999326	0.999312	0.999299	0.999285	0.999272	0.999258
14	0.999244	0.999230	0.999216	0.999202	0.999188	0.999173	0.999159	0.999144	0.999129	0.999114
15	0.999099	0.999084	0.999069	0.999054	0.999038	0.999023	0.999007	0.998991	0.998975	0.998959
16	0.998943	0.998926	0.998910	0.998893	0.998877	0.998860	0.998843	0.998826	0.998809	0.998792
17	0.998774	0.998757	0.998739	0.998722	0.998704	0.998686	0.998668	0.998650	0.998632	0.998613
18	0.998595	0.998576	0.998558	0.998539	0.998520	0.998501	0.998482	0.998463	0.998444	0.998424
19	0.998405	0.998385	0.998365	0.998345	0.998325	0.998305	0.998285	0.998265	0.998244	0.998224
20	0.998203	0.998183	0.998162	0.998141	0.998120	0.998099	0.998078	0.998056	0.998035	0.998013
21	0.997992	0.997970	0.997948	0.997926	0.997904	0.997882	0.997860	0.997837	0.997815	0.997792
22	0.997770	0.997747	0.997724	0.997701	0.997678	0.997655	0.997632	0.997608	0.997585	0.997561
23	0.997538	0.997514	0.997490	0.997466	0.997442	0.997418	0.997394	0.997369	0.997345	0.997320
24	0.997296	0.997271	0.997246	0.997221	0.997196	0.997171	0.997146	0.997120	0.997095	0.997069
25	0.997044	0.997018	0.996992	0.996967	0.996941	0.996914	0.996888	0.996862	0.996836	0.996809
26	0.996783	0.996756	0.996729	0.996703	0.996676	0.996649	0.996621	0.996594	0.996567	0.996540
27	0.996512	0.996485	0.996457	0.996429	0.996401	0.996373	0.996345	0.996317	0.996289	0.996261
28	0.996232	0.996204	0.996175	0.996147	0.996118	0.996089	0.996060	0.996031	0.996002	0.995973
29	0.995944	0.995914	0.995885	0.995855	0.995826	0.995796	0.995766	0.995736	0.995706	0.995676
30	0.995646	0.995616	0.995586	0.995555	0.995525	0.995494	0.995464	0.995433	0.995402	0.995371

Compare the theoretical density with the average of the experimentally measured densities for each piece of measuring apparatus. Then calculate the absolute error (difference between the average and theoretical densities) involved for each apparatus. The formula for this calculation is:

$$Absolute\ error = |measured\ value - theoretical\ value|$$

Since the value is an "absolute" value, the sign will be positive to reflect the difference in the two values. The smaller the absolute error, the more accurate the measurement. In the conclusion, rank the glassware in order of least to most accurate based on YOUR data.

Report: Complete the following report form neatly and using complete sentences. This will be due the next time lab meets. A typed lab report is not required.

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Accuracy and Precision Lab Report Form

Accepted 5pts / Accepted 3pts / Rejected 0pts

Prelab (1 pt) _____ Name_____

DATA: Partner_____

Part A

Water Temp:_____

Theoretical density of water_____g/mL (from the table and the temperature)

Predict which piece of glassware you expect to be the most accurate and precise as well as the least accurate and precise. Explain why you made these predictions:

Most accurate:_____ why?_____

Least accurate:_____ why?_____

Most precise:_____ why?_____

Least precise:_____ why?_____

Graduated Cylinder:

	Volume (mL) recorded to 1 decimal place	Mass water (g)	Density (g/mL) calculated
Trial 1			
Trial 2			
Trial 3			

Pipette:

	Volume (mL)	Mass water (g)	Density (g/mL) calculated
Trial 1	10.00		
Trial 2	10.00		
Trial 3	10.00		

Burette:

	Initial Volume (mL) measured to 2 decimal places	Final Volume (mL) measured	Volume (mL) calculated	Mass water (g)	Density (g/mL) calculated
Trial 1					
Trial 2					
Trial 3					

Calculations and Results:

Show all calculations with units for the graduated cylinder and report results for all trials and glassware to the correct number of significant figures in the results table.

Graduated Cylinder Calculations:

Density of water calculation for trial 1 only:

Density Range:

Average Density:

Absolute Error:

RESULTS TABLE

Glassware	Density Range	Average Density	Absolute Error
Grad. Cylinder			
Burette			
Pipette			

Discussion and Conclusion:

Rank the types of glassware in order of least to most precise. DISCUSS in complete sentences what data (numbers) led you to draw these conclusions.

Rank the types of glassware in order of least to most accurate. DISCUSS in complete sentences what data (numbers) led you to draw these conclusions.

Error:

1. Sources of error. **<u>For each lab going forward</u>**, you will need to identify and discuss sources of error. Answer the questions below to learn what types of error we are looking for and what information to provide now and in the future each time you make a measurement.

    a. How is the volume in a graduated cylinder measured? (What do you physically DO to measure it?)

    b. Given how it is measured, what causes variability in this measurement?

    c. Does this error always cause a high or low value? Is the error random or systemic?

    d. How does the error in this measurement effect the final result? (In this case, how does a high/low value effect the calculated density, and then ultimately the conclusions you drew about the glassware rankings?

    e. What, if anything, can you do to reduce this error?

Pre-Laboratory Assignment for: Percent Yield - Synthesis of Alum: $KAl(SO_4)_2 \bullet 12H_2O$

Write the questions and answers in your duplicating NB.

1. Vocabulary: (Please define or describe the following)
    a. Corrosive –
    b. Buchner Funnel –
    c. Synthesis –
    d. Theoretical Yield -
    e. Percent Yield –

2. Read the lab. In your own words and in complete sentences:
    a. Describe the purpose of the lab. Why are we doing it?
    b. What concepts and/or calculations will we be using?

3. In a table, list the glassware and equipment we will be using and their purpose/function. For illustration the first is done for you:

Glassware/Equipment	Purpose/Function
250mL beaker	Vessel to perform the reaction in.

4. In a table, list the chemicals and other potentially hazardous equipment/procedures we will be using in THIS lab and how you are going to minimize your risks.
For illustration the first few are done for you:

CHEMICAL/EQUIPMENT/PROCEDURE	HAZARD AND MY STEPS TO STAY SAFE
1. Burn from hot plate	I will keep all materials away from the hot plate including the cord. I will not touch the surface of the hot plate even if I think it is cool.
2. Potassium Hydroxide	Can cause burns – caustic. I will wear gloves, goggles and a lab coat. I will wash my hands often.
3.	

Percent Yield - Synthesis of Alum: $KAl(SO_4)_2 \bullet 12H_2O$

Objective:
To use aluminum foil to synthesize a chemical compound, alum, which is hydrated potassium aluminum sulfate, $KAl(SO_4)_2 \bullet 12H_2O$.

Hazards:
- Potassium hydroxide is a strong, corrosive base. If you get in on you your skin will feel slippery, rinse with lots of water.
- Sulfuric acid is a strong, corrosive acid. Wear gloves. If you get it on your skin it will burn, rinse with lots of water.
- You will be generating H_2 gas (hydrogen gas), it is VERY flammable, do this step in the HOOD!
- Wear goggles and a lab coat/apron at all times!

Introduction:
Alum is a very versatile compound; its many uses include use as a flocculate (clarifier) in water purification processes, in fire extinguishers, as an astringent, or as an ingredient in baking powder. You will take a clean piece of aluminum foil and dissolve it in a potassium hydroxide solution to create a complex ion called "aluminate" $[Al(OH)_4^-]$. This can be described by the following complete and net ionic equations:

$$2Al_{(s)} + 2KOH_{(aq)} + 6H_2O_{(l)} \rightarrow 2KAl(OH)_{4(aq)} + 3H_{2(g)}$$
$$2Al_{(s)} + 2OH^-_{(aq)} + 6H_2O_{(l)} \rightarrow 2Al(OH)_4^-{}_{(aq)} + 3H_{2(g)}$$

After filtration to remove any unwanted material, the alkaline solution of $Al(OH)_4^-$ is ***clear*** and ***colorless***. H_2 gas is evolved, which mixes with the atmosphere. The chemical species that remain in solution are potassium ions (K^+) and aluminate ions $[Al(OH)_4^-]$, plus any unreacted potassium hydroxide (KOH).

In the next step, sulfuric acid is added and two sequential reactions occur. Initially, before the addition of acid is complete, insoluble aluminum hydroxide is formed $[Al(OH)_{3(s)}]$. It appears as a ***thick, white, gelatinous precipitate***. This reaction is described by:

$$2KAl(OH)_{4(aq)} + H_2SO_{4(aq)} \rightarrow 2Al(OH)_{3(s)} + 2H_2O_{(l)} + K_2SO_{4(aq)}$$

As more sulfuric acid is added, the precipitate of $Al(OH)_3$ dissolves to form soluble Al^{3+} ions, according to the following fully balanced equation:

$$2Al(OH)_{3(s)} + 3H_2SO_{4(aq)} \rightarrow Al_2(SO_4)_{3(aq)} + 6H_2O_{(l)}$$

At this point, the solution contains Al^{3+} ions, K^+ ions, and SO_4^{2-} ions. On cooling, crystals of hydrated potassium aluminum sulfate, $KAl(SO_4)_2 \bullet 12H_2O$ (or alum) are very slowly deposited. The crystallization process can be sped up by scratching the inside of the flask or by providing a small "seed crystal" of alum for the newly forming crystals to grow on. Cooling is needed because alum crystals are soluble in water at room temperature.
(Can you think of something that could decrease your yield? Record this as a source of error!)

This occurs in several steps, but the full equation for the formation of alum is below:

$$2Al_{(s)} + 2KOH + 4\ H_2SO_{4(aq)} + 22H_2O_{(l)} \rightarrow\ 2KAl(SO_4)_2 \bullet 12H_2O_{(s)} + 3H_{2(g)}$$

The mole to mole ratio of Al to *$KAl(SO_4)_2 \bullet 12H_2O_{(s)}$* is 1:1. You will need this to calculate your theoretical yield. Finally, the crystals are removed by vacuum filtration and washed with an alcohol/water mixture. The wash liquid removes any contamination from the crystals but does not dissolve them. It also helps to dry the crystals quickly, because alcohol is more volatile than water.

Your goal is to make the alum compound, then determine your percent yield. Careful attention to measurements and how you handle materials will help ensure you get the maximum possible yield. This is part of your grade!

Procedure: WEAR GOGGLES, GLOVES and an APRON/LAB COAT

I. Reaction of aluminum with KOH (the dissolution step)

$$2Al_{(s)} + 2KOH_{(aq)} + 6H_2O_{(l)} \rightarrow 2KAl(OH)_{4(aq)} + 3H_{2(g)}$$

NIE: $2Al_{(s)} + 2OH^-_{(aq)} + 6H_2O_{(l)} \rightarrow 2Al(OH)_4^-{}_{(aq)} + 3H_{2(g)}$

$Al(OH)_4^-{}_{(aq)}$ is the aluminate ion

Before you start: Make sure all glassware is clean and dry before beginning this experiment.

As with ALL labs, throughout the procedure, **record observations**. These are things you can detect with your senses. Also note the steps that contribute to loss of material and will reduce your yield**. Record these as sources of error/loss.**

1. Place a 250 mL beaker (labeled with your names) on the balance and tare. Obtain a piece of aluminum foil and cut it into small pieces. Add them to the beaker until you have added about 1 g.

2. Record the exact mass (ALL numbers given on the balance – NEVER round) of your foil in your duplicating notebook.

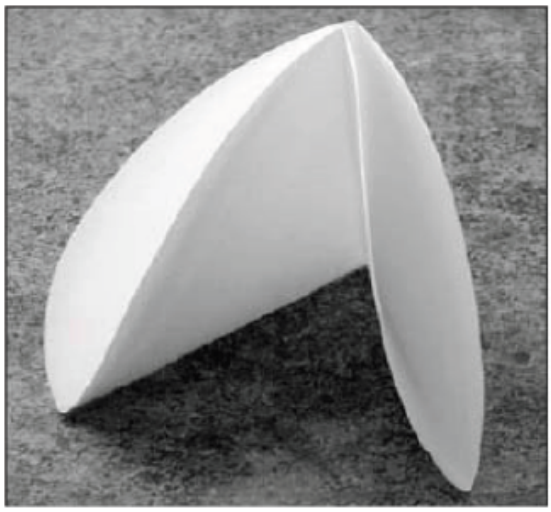
3. DO THIS STEP IN THE HOOD! Using a graduated cylinder, add 50mL of 1.4M KOH to the beaker and leave in the hood to react.

4. The aluminum will take about 10-15 minutes to dissolve. Push any aluminum off the sides back into the solution with your glass stirring rod.

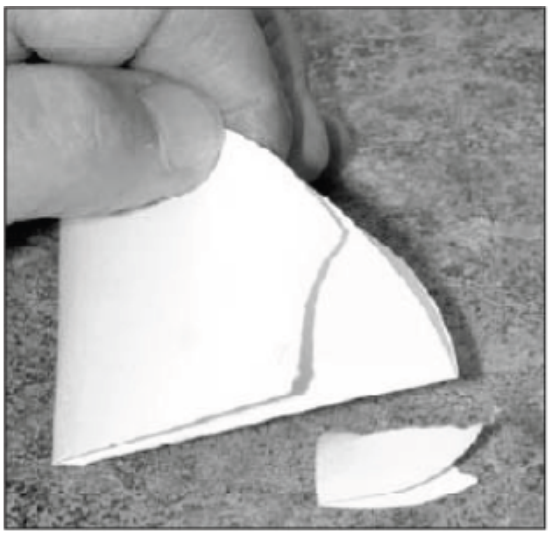
   PLAN AHEAD: While the aluminum is dissolving, set up an apparatus for **gravity filtration** (see figure below). Place a clean funnel with the piece of folded filter paper on a 125 mL Erlenmeyer flask.

Preparing a Gravity Filtration

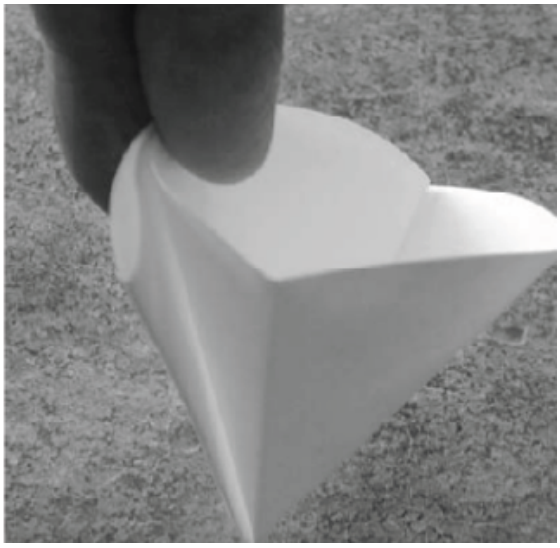
Filtration is used to separate a solid from a mixture of that solid suspended in a liquid or a solution. The simplest filtration method used is to pour the mixture through a filter. The solids get stuck in the filter and the liquid phase passes through. In order to obtain as pure a solid as possible, the solid is washed with pure solvent.



Step 1: Fold the filter paper in half, and then in half again



Step 2: It helps the filter work more effectively if you tear off a small corner. If you are going to weigh the paper, make sure you do so AFTER you tear off the corner.



Step 3: Separate the top edges of the paper so 3 go one way and the fourth in the opposite direction.

Step 4: Place the filter paper in the funnel. Moisten the paper with a few drops of distilled water from your squeeze bottle.

5. When the aluminum has dissolved (as evidenced by the lack of H_2 bubbles given off), bring the beaker back to your station. If Aluminum remains after 10 minutes, heat on a hot-plate on low setting.

6. Gravity filter the solution. Only fill the funnel to within ½ an inch from the top of the paper. Use a glass stirring rod to guide the solution into the paper (as demonstrated by your instructor). The solution in the bottom of the Erlenmeyer flask should be both clear and colorless. If it is not, filter again through a fresh piece of filter paper.

   PLAN AHEAD: While the solution is filtering, calculate the theoretical yield from the mass of aluminum you started with. See "calculations".

7. Rinse the beaker and rod with a small amount of purified water and add it to the filter paper to lose as little aluminum ions as possible.

8. When the solution has passed through the paper, rinse the filter paper with a very **small** amount of purified water using your squeeze bottle and allow it to pass through the filter. Don't use too much water as this will delay the formation of the crystals.

9. Allow the flask to cool. While cooling, wash the funnel, beaker and filter paper with lots of tap water to remove any KOH. The rinsed filter paper can go in the garbage.

II: Initial addition of sulfuric acid, H_2SO_4 (precipitation of $Al(OH)_3$)

$2\ KAl(OH)_{4(aq)} + H_2SO_{4(aq)} \rightarrow 2\ Al(OH)_{3(s)} + 2H_2O_{(l)} + K_2SO_{4(aq)}$

III: Further addition of sulfuric acid, H_2SO_4 (dissolving of $Al(OH)_3$)

$2\ Al(OH)_{3(s)} + 3\ H_2SO_{4(aq)} \rightarrow Al_2(SO_4)_{3(aq)} + 6\ H_2O_{(l)}$

IV: Precipitation of alum on cooling (alum crystals formation)

$K_2SO_{4(aq)} + Al_2(SO_4)_{3\,(aq)} + 24\ H_2O_{(l)} \rightarrow 2\ KAl(SO_4)_2 \bullet 12H_2O_{(s)}$

The overall net ionic equation for the precipitation process:

$K^+_{(aq)} + Al^{3+}_{(aq)} + 2\ SO_4^{2-}{}_{(aq)} + 12\ H_2O_{(l)} \rightarrow KAl(SO_4)_2 \bullet 12\ H_2O_{(s)}$

10. When the solution is reasonably cool, add 20 mL of 9M H_2SO_4 (in the hood), quickly and with care.
NOTE: It is important that you swirl the flask as you add the acid. The solution will get quite warm. If there are any white flecks left in the solution after the addition of the H_2SO_4, place the flask on a hot-plate and warm it gently while swirling until all solid has been dissolved. If a large amount of white precipitate remains, add an additional 5 mL of acid and swirl.

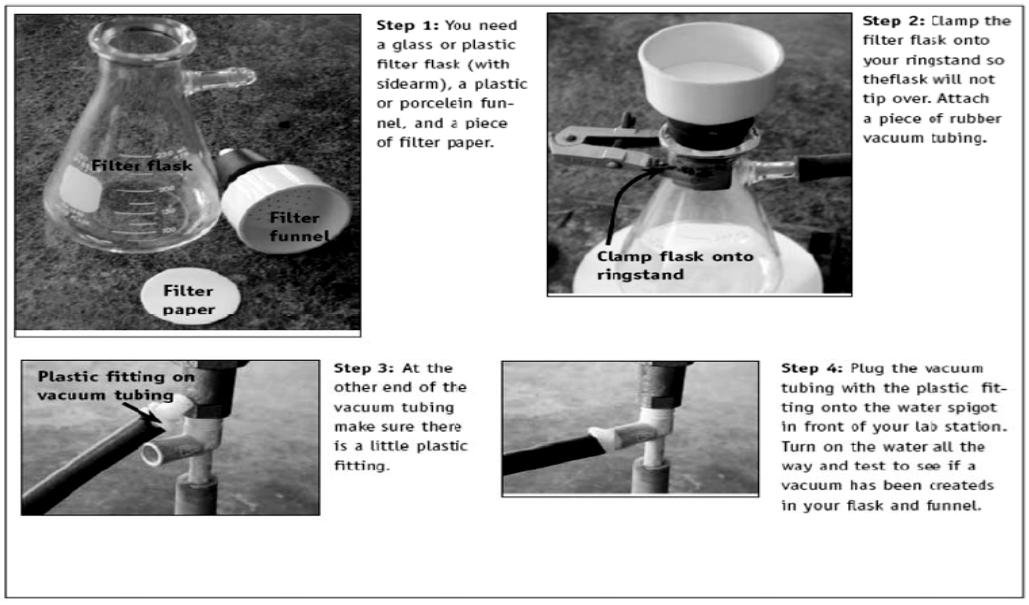
PLAN AHEAD: Make an ice bath by putting ice and a little water in a 600 mL beaker.

11. Allow the flask to cool until just warm, then put it in the ice bath for at least 5 minutes.

PLAN AHEAD: While the solution is cooling, pour 50 mL of the 50% alcohol/water mixture into a few test tubes or a clean graduated cylinder and cool it in an ice bath. Take this time to answer the questions at the end of the lab report form.

12. If alum crystals have not started to form, scratch the inside wall of the flask with a stirring rod. This provides a site that the crystals can latch onto and begin crystallization. Swirl liquid gently once crystals start to form, then let it sit in the ice bath for 10 – 15 minutes undisturbed.

PLAN AHEAD: Record the mass of a small piece of filter paper. Set up a **vacuum filtration** apparatus (Buchner funnel, see figure below) and as demonstrated by your instructor. Spray a small amount of purified water on the filter paper using your squeeze bottle with the vacuum on. Make sure that all of the holes in the Buchner funnel are covered by the filter paper.



13. Remove the flask containing the alum crystals from the ice bath, swirl so that the crystals are dislodged, and pour quickly into the Buchner funnel. Keep swirling and pouring until all of the solution and crystals are transferred to the funnel – keep the aspirator on at all times during this process. If filtrate is cloudy, chill in ice for another 5-10 minutes and re-filter through the same filter paper.
14. Pour about 10 mL of the chilled alcohol/water mixture into the flask that contained the crystals. Swirls and pour into the Buchner funnel. Repeat until all of the mixture has been used.
15. When done filtering, place your crystals and filter paper in a clean dry 50 mL beaker, label them and store them in your drawer or other designated area until next week to ensure they are completely dry.
    PLAN AHEAD: Weigh the dry 50 mL beaker and record its mass in your notebook along with the mass of the vacuum filtration filter paper you recorded earlier. Having these masses together will allow you to determine the mass of your final product more easily next week. Mass of Final Product = (mass of beaker + filter paper + product) – (mass of beaker + filter paper)
16. Scrub all glassware and the Buchner funnel, and then return any borrowed supplies to the appropriate part of the lab.
17. Complete all parts of the report form except what needs to be completed next week. Be neat and use complete sentences.

Next Week:

Weigh your alum crystals, record their appearance, complete the calculation for percent yield using the actual mass product, and complete your discussion**. Put the dried alum in the designated container.**

Calculations:

Theoretical yield in grams of Alum may be calculated now. You need to use the given fact that the mole/mole ratio of aluminum to alum is 1:1. The molar mass for 1 mole of aluminum can be read directly from the periodic table. The molar mass for Alum must be calculated. Add the masses from the periodic table for each atom in the formula (the formula for alum is in the title of this lab). You must account for numbers of each atom. For example: $CaCl_2$'s molar mass is obtained by adding the mass of 1 mole of calcium and 2 moles of chlorine: 40.078 + (2* 35.4527) = 110.983 g/mol

Now: Calculate theoretical yield with the following conversions
- mass Al to moles Al
- moles Al to moles alum (1:1)
- moles alum to mass alum

Next week: Percent yield = (actual yield/theoretical yield) *100%

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Alum Lab Report Form

Name_____

Prelab 1 pt _____ Partner_____

Accept 5 pts / Accept 3 pts / Reject 0 pts

DATA:

Mass Al:_____g_

Observations through gravity filtration:

Mass filter paper: _____g__

Observations from gravity filtration through vacuum filtration:

Mass dry alum, beaker and filter paper (week 2): _____g_

Observations of dry crystals:

Calculations and Results:

Show all calculations with units and report results to the correct number of significant figures in the table.

Actual yield (subtract beaker and filter paper):

Theoretical yield:

% Yield:

RESULTS TABLE

Actual Yield	Theoretical Yield	% Yield

Discussion and Conclusion:

The discussion below is scaffolded for you to help you see what a good discussion looks like.

The compound Alum with a formula of _____ was synthesized by the following reaction_____. The appearance of the final product was _____. Gravity filtration was used during the process to remove contaminants from the aluminum and potassium ions and vacuum filtration was used to isolate and clean the crystals of final product.

From _____g of aluminum foil, the theoretical yield of Alum is _____g. The actual yield obtained was _____g, therefore the percent yield from this synthesis was _____%. There were several steps that could have led to loss which resulted in a lower percent yield. I will describe three and suggest ways to reduce the loss:

Questions:

1. Describe the equipment and purpose of both gravity filtration and vacuum filtration and include a drawing of each apparatus. Then list at least 3 similarities and differences between them.

   Gravity Filtration:

   Vacuum Filtration:

   Similarities:

   Differences:

2. Students often confuse percent error and percent yield.

    a. What is the formula for percent error?

    b. What is percent error used for?

    c. Explain the difference between percent yield and percent error.

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Pre-Laboratory Assignment for: Reaction Stoichiometry – Determining a Limiting Reactant

Write the questions and answers in your duplicating NB.

1. Vocabulary: (Please define or describe the following)
    a. Limiting Reactant –
    b. Percent Difference –
    c. Decomposition –

2. Read the lab. In your own words and in complete sentences:
    a. Describe the purpose of the lab. Why are we doing it?
    b. What concepts and/or calculations will we be using?

3. In a table, list the glassware and equipment we will be using and their purpose/function. For illustration the first is done for you:

Glassware/Equipment	Purpose/Function
50mL beaker	Vessel to perform the reaction in.

4. In a table, list the chemicals and other potentially hazardous equipment/procedures we will be using in THIS lab and how you are going to minimize your risks.

CHEMICAL/EQUIPMENT/PROCEDURE	HAZARD AND MY STEPS TO STAY SAFE
1.	
2.	
3.	

Reaction Stoichiometry – Determining a Limiting Reactant

Alka-Seltzer tablets contain sodium bicarbonate, aspirin, and citric acid. When the tablet is dissolved in water the sodium bicarbonate reacts with the acids to form carbonic acid which quickly decomposes into carbon dioxide and water.

In most chemical reactions, the reactants are combined in amounts that are not a perfect ratio to all fully react to products. Therefore, one reactant "runs out" first and the other reactants are in excess. These are called limiting reactant (or reagent) reactions.

Objective:

In this experiment, you will combine sodium bicarbonate and citric acid in varying amounts and calculate which reactant is fully consumed and stops the reaction. In other words, you will determine which one is the limiting reactant. You will also determine the amount of excess reactant remaining and devise an experiment to test your results.

The UNBALANCED reaction is given below.

$$\mathbf{NaHCO_{3(aq)} + H_3C_6H_5O_{7(aq)} \rightarrow H_2O_{(l)} + CO_{2(g)} + Na_3C_6H_5O_{7(aq)}}$$

Since the product carbon dioxide is a gas and we will be working in an "open system," it will be lost to the atmosphere during the reaction. Therefore the difference in mass of everything before the reaction and after the reaction will equal to the mass of the lost carbon dioxide. [beaker and reactants – beaker and products = CO_2 lost] You will use this mass, the balanced equation and stoichiometry to determine the amount of each reactant that actually reacted. Comparing these results to the masses physically measured will enable you to determine the limiting reactant as well as the mass of the excess reactant.

Copy the following into your notebook and be sure to include it in your report. Make the table large in your notebook so you have enough room to make it NEAT.

Data and Observations:

(Record ALL numbers displayed on the balance)	Trial 1	Trial 2
Dry beaker (g)		
Water (g)		
$NaHCO_3$ (g)		
Weigh boat and $H_3C_6H_5O_7$, citric acid (g)		
"Empty/Dirty" weigh boat after addition of citric acid		
$H_3C_6H_5O_7$, citric acid (g) Subtract the 2 lines above.		
Total before the reaction (add beaker + water + both reactants)		
Total mass after the reaction		
Mass CO_2 "Actual Yield" (total before – total after)		

Observations: (Always record observations in your notebook as part of your raw data.)

(Leave space in your notebook to record your observations)

Procedure

Trial 1

1. Clean and dry a 50 mL beaker. Bring the beaker, your notebook and a graduated cylinder with 10ml purified water to the balance with you. Record the mass of the empty beaker in your NB.
2. Tare the beaker and add 10 mL purified water. Record the mass of the water.
3. Tare the beaker and the water and add between 1 and 2 grams of sodium bicarbonate to the beaker. Record the mass in your NB.
4. Tare the empty balance. Place a weigh boat on the pan and measure between 1 and 2 grams of citric acid. Record the mass of (the total of both together) weigh boat AND the citric acid in your NB.
5. Slowly add the citric acid to the beaker.
6. Swirl gently until the reaction is complete and record your observations.
7. Tare the SAME empty balance and record the mass of the "empty/dirty" weigh boat. Subtract to determine the exact amount of citric acid that was added to the beaker.
8. While waiting for the reaction to complete, in your NB, calculate which reactant was the limiting reactant based on YOUR reactant masses. Calculate the amount (g) of excess reactant that remains. Transfer these calculations neatly to your report form and complete what you can of the report form while your reaction occurs.
9. Swirl to remove bubbles and record the final mass of the beaker and its contents.
10. DO NOT DISCARD the contents.
11. Devise an experiment to test your conclusion, (which reactant is limiting) using only the available materials and your completed reaction.
12. Record this "confirmation procedure" in your NB. Show and get approval from your instructor before using the procedure.
13. Perform your confirmation experiment and record your observations.
14. Before moving on, check your calculations with your instructor and repeat Trial 1 with similar masses if the results of your confirmation experiment differ from what you expected.

Trial 2

15. Discard your reaction for Trial 1 down the drain. Clean and DRY the beaker.
16. From your calculated results in part 1, select masses for an additional trial that will make the **other** reactant limiting (not the reactant that was limiting in Trial 1). Be careful, if you choose too small of an amount of one reactant you will not generate enough CO_2 to detect a change in final mass.
17. Calculate the mass of carbon dioxide you expect to generate. This is your theoretical yield.
18. Perform the experiment and record your data.
19. Record the mass of carbon dioxide produced as "actual yield." This is the difference between the beaker plus reactants and the beaker plus products.
20. Repeat all calculations to determine the limiting reactant and the mass of the excess reactant that will remain following the reaction.
21. Perform and record observations for the confirmation procedure. (Will it be the same? Be careful!)
22. Clean and return your glassware. All solutions may go down the drain.
23. Calculate the percent error between your actual yield of CO_2 and the theoretical yield for both trials.

$$\% \text{ error} = \frac{|\text{actual} - \text{theoretical}|}{\text{theoretical}} * 100\%$$

24. NEATLY transcribe all of your data and calculations to the report form. Complete the other portions of the report form with complete sentences.

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Limiting Reactant Lab Report Form

Name_____

Partner_____

Prelab (1 pt) _____

Accept 5 pts / Accept 3 pts / Reject 0 pts

PROCEDURE: Briefly summarize the experimental procedure as if you are describing it to a non-scientist. Explain what you did and why. (Do not list the steps) Include the confirmation test procedure you developed.

Data and Observations:

(Record ALL numbers displayed on the balance)	Trial 1	Trial 2
Dry beaker (g)		
Water (g)		
$NaHCO_3$ (g)		
Weigh boat and $H_3C_6H_5O_7$, citric acid (g)		
"Empty/Dirty" weigh boat after addition of citric acid		
$H_3C_6H_5O_7$, citric acid (g) Subtract the 2 lines above.		
Total before the reaction (add beaker + water + both reactants)		
Total mass after the reaction		
Mass CO_2 "Actual Yield" (total before – total after)		

Observations:

Calculations and Results:

Show all calculations with units and report results rounded to the correct number of significant figures in the table.

Write the balanced equation: _____

Trial 1

Actual Yield of Carbon Dioxide Generated Calculation

Theoretical carbon dioxide calculation for EACH reactant. Circle the limiting reactant.

% Error Calculation

Limiting Reactant Identity_____

Excess Reactant Identity_____

Mass of Excess Reagent Calculation

Trial 2

Actual Yield of Carbon Dioxide Generated Calculation

Theoretical carbon dioxide calculation for EACH reactant. Circle the limiting reactant.

% Error Calculation

Limiting Reactant Compound_____

Excess Reagent Compound_____

Excess Reagent Calculation

Calculated Results:

	Trial 1	Trial 2
Actual Yield of CO_2 (g)		
Theoretical Yield of CO_2 (g) if $NaHCO_3$ mass is used		
Theoretical Yield of CO_2 (g) if $H_3C_6H_5O_7$ mass is used		
Name of Limiting Reactant (LR)		
Result of Confirmation Test (was LR prediction correct?)		
Percent Error of Actual to Theoretical mass of CO_2		
Excess reactant mass (g)		

Question:

1. One of the indications of Alka-Seltzer is that it will neutralize stomach acid. When the tablet is dissolved in water before consumption, which reactant do you think is the limiting reactant in the actual medication? EXPLAIN your answer.

Discussion and Conclusion:

Discuss the percent error between the theoretical yield and your actual yield for each trial. Was your actual yield higher or lower than your theoretical? Include three sources of error that could have led to this discrepancy. Do NOT write "human error." If you know you made a mistake, you should have repeated the experiment! Instead, give specific examples of unavoidable errors inherent in the procedure and measurements. Keep in mind that losses of reactants or solution will actually lead to the error that you think you have produced MORE carbon dioxide than you really did. Explain what this did to your % yield?

Pre-Laboratory Assignment for: Solution Stoichiometry - Determination of the Molar Mass of an Unknown Diprotic Acid

1. Vocabulary: (Please define or describe the following)
    a. Titration –
    b. Standardization (of a solution) –
    c. Acid-base equivalence point –
2. Read the lab. In your own words and in complete sentences:
    a. Describe the purpose of the lab. Why are we doing it?
    b. What concepts and/or calculations will we be using?
3. In a table, list the glassware and equipment we will be using and their purpose/function.

Glassware/Equipment	Purpose/Function
1.	
2.	

4. In a table, list the chemicals and other potentially hazardous equipment/procedures we will be using in THIS lab and how you are going to minimize your risks.

CHEMICAL/EQUIPMENT/PROCEDURE	HAZARD AND MY STEPS TO STAY SAFE
1.	

5. For each $H_2C_2O_4 \bullet 2H_2O$ formula unit, how many NaOH formula units will titrate with it? This is the same as the mol:mol ratio! Write the balanced equation for the titration.

6. As part of the experimental procedure, you will "place approximately 250 mL of distilled water into a 500 mL Erlenmeyer flask. Add approximately 50 mL of 3M NaOH. NOTE – the total amount of solution is now 300 mL!"
Determine the approximate concentration of the NaOH solution you will prepare. (Hint: You can use the quick formula $M_1V_1 = M_2V_2$ or dimensional analysis)

Solution Stoichiometry: Determination of the Molar Mass of an Unknown Diprotic Acid

Introduction:

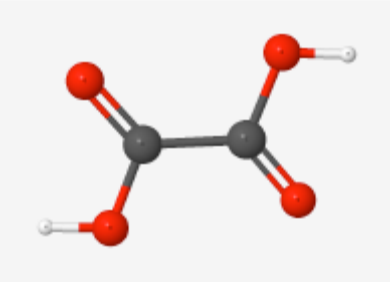
Titration is the process for ascertaining the exact volume of one solution that is chemically equivalent to a given amount of another substance, either another solution or a given amount of solid material dissolved in a solvent. In other words, this is NOT a limiting reactant reaction. In titration, we start with a measured amount of one reactant and add just enough of the other reactant until the reaction is complete. The apparatus usually used in titrations is a burette. If a solution of an acid is titrated with a solution of a base, the **equivalence point**, the point at which the reaction is complete, can be seen by a color change of another added chemical called an indicator.

In this lab, you will be standardizing (determine precisely the concentration of) a solution of a base sodium hydroxide, NaOH, using oxalic acid dihydrate, $H_2C_2O_4 \bullet 2H_2O$, as a primary standard acid. Then you will use the sodium hydroxide, with it's now known concentration, to titrate an unknown acid, and then use the titration information to solve for the acid's molar mass.

Hazards:

Sodium hydroxide is caustic. If you get it on your skin, it will feel slippery; rinse it off with lots of water.

In this experiment, you must first standardize the base, NaOH, with a primary standard acid, $H_2C_2O_4 \bullet 2H_2O$. A primary standard acid is a solid acid whose mass is an accurate measure of the number of moles of H^+ ions it will furnish.



Oxalic acid, $H_2C_2O_4$, has two ionizable H atoms, so two moles of NaOH are required to consume one mole of acid.

The balanced equation for the acid-base reaction involved in the standardization procedure is:

$$H_2C_2O_4 \bullet 2H_2O_{(aq)} + 2NaOH_{(aq)} \rightarrow Na_2C_2O_{4(aq)} + 4H_2O_{(l)}$$

This equation specifies that there are two moles of H^+ supplied by each mole of oxalic acid in this reaction (since 1 mole of H^+ is consumed per mole of NaOH). From the mass of the oxalic acid dihydrate used in the reaction, you can calculate the moles of acid used. This is related to the amount of NaOH

known and the volume of solution in which these are contained; you can calculate the concentration of the NaOH (moles of NaOH/ liters of solution). That is, you have now standardized the solution of NaOH and can use it to determine the molar mass of an unknown, diprotic acid.

In the experiment you will be given a sample of unknown acid. You only know that it reacts with NaOH according to the general equation:

$$H_2A_{(aq)} + 2NaOH_{(aq)} \rightarrow Na_2A_{(aq)} + 2H_2O_{(l)}$$

The acid is known to supply TWO moles of H^+ per mole of acid. When you titrate a weighed sample of the unknown acid with standardized NaOH, you will determine the volume of NaOH required. Therefore, you know the quantity of NaOH (mole) used from the relationship:

Moles of NaOH = Concentration of NaOH (mol/L) x Volume of NaOH (L)

The moles of NaOH consumed are related to the moles of the unknown diprotic acid through stoichiometric factor (of ½, based on the balanced equation) to the moles of acid that were contained in the sample.

$$\text{Moles of Unknown Acid} = \text{Moles of NaOH Used} \times \frac{1\text{mol acid}}{2\text{mol NaOH}}$$

Finally, the molar mass of the acid can be found from the relation:

$$\text{Molar Mass of Acid (g/mol)} = \frac{\text{Grams of Acid Sample}}{\text{Moles of Acid Sample}}$$

Procedure:

Goals of the experiment:

1. Prepare and standardize a NaOH solution
2. Use the standardized solution to titrate an unknown acid
3. Determine the molar mass of the unknown from your data

Part A Standardization of a Solution of Sodium Hydroxide:

1. Clean and rinse your graduated cylinder, flasks, and beakers. Clean a burette and rinse it with distilled water until the water drains cleanly from the inverted burette.
2. Prepare a dilute solution of sodium hydroxide by placing approximately 250ml of distilled water into a 600 mL Erlenmeyer flask. Add approximately 50 mL of 3M NaOH (NOTE – the total amount of solution is now 300 mL!) Swirl to mix well.
3. Rinse your burette a few times with small (5 mL) portions of your dilute NaOH solution. Drain each rinse into a waste beaker, label the beaker as waste. Fill the burette nearly to the top of the graduated portion with the NaOH solution and make sure that the burette tip is full of solution. Read the initial burette reading to 2 decimal places (estimate the last digit).
4. Assuming you would like to use about 25 mL of your ~0.5M NaOH solution, calculate the mass (g) of oxalic acid dihydrate needed to standardize the NaOH. Weigh out the appropriate amount of oxalic acid and record all decimal places.

5. Place the sample in a clean 125 mL Erlenmeyer flask, add about 50 mL of distilled water. Rinse all the solid off the inside of the flask into the solution with your squeeze bottle. Swirl flask but it may not all dissolve before titrating. Add five drops of phenolphthalein indicator. Place your Erlenmeyer flask with the acid in it under the burette with a white piece of paper underneath. Begin the titration by adding sodium hydroxide to the oxalic acid solution while swirling until the endpoint is reached. When it reaches its endpoint, the solution *just* turns from colorless to pink – this will likely happen with a single drop of NaOH solution. When pink color begins to persist for longer periods of time, begin to add NaOH dropwise to avoid exceeding the endpoint. Record the final burette reading to 2 decimal places.
6. Titrate 2 more samples of the standard acid (total of 3), being certain that the burette is refilled nearly to the top of the graduated portion with the NaOH solution and use a clean flask for the new sample.

DO NOT WASTE THE NaOH SOLUTION!! You will use the NaOH you just standardized to determine the molar mass of an unknown acid. If you waste or throw it away you will have to start from the beginning again!

Part B Determination of the Molar Mass of an Unknown, Diprotic Acid

1. Obtain an unknown acid and record the number in your notebook.

Prepare 3 SEPARATE samples of the unknown to be titrated as 3 separate trials:

2. Label three weigh boats as 1, 2 and 3. Weigh three, 1 gram each, samples of the same unknown acid. Record the masses as Part B unknown acid mass Trials 1-3.
3. Label three 125 mL Erlenmeyer flasks. Add each sample, about 50 mL of distilled water and 5 drops of phenolphthalein to three different 125 mL Erlenmeyer flasks just like you did for part A.
4. Titrate the samples one at a time with the standardized NaOH solution to a permanent light pink endpoint. Your unknown sample may not be completely dissolved when you start the titration, but the dissolution process will continue as you add NaOH; make sure the entire solid is dissolved before you record the endpoint.

Suggested Data Table. RECORD YOUR UNKNOWN ACID NUMBER/LETTER

Part A	Oxalic acid (g)	Initial Burette (mL)	Final Burette (mL)	NaOH used (mL)
Trial 1				
Trial 2				
Trial 3				
Leave space for additional trials if needed				
Part B	**Unknown # "_" (g)**	**Initial Burette (mL)**	**Final Burette (mL)**	**NaOH used (mL)**
Trial 1				
Trial 2				
Trial 3				

Part A Calculations:
Standardization: find the "exact" concentration of NaOH solution
$H_2C_2O_4 \bullet 2H_2O_{(aq)} + 2NaOH_{(aq)} \rightarrow Na_2C_2O_{4(aq)} + 4H_2O_{(l)}$
2 moles NaOH are needed to neutralize 1 mole of diprotic acid!
For EACH trial, use g acid to find moles acid.
Use moles acid to find moles base (balanced equation)
Divide the moles base by the liters of base = M base
Average the trials that agree (use your best judgment)

Part B Calculations:
$H_2A_{(aq)} + 2NaOH_{(aq)} \rightarrow Na_2A_{(aq)} + 2H_2O_{(l)}$
Use liters of base used from the burette (final – initial) and the average molarity of base calculated in part A: (L base) x (molarity base) = moles base
Moles base * (1 mol acid/2 moles base) = moles unknown diprotic acid
Divide the grams of unknown acid you weighed out by the moles of acid you just calculated.
Grams unknown acid/moles unknown acid = molar mass g/mol !!
Average the trials that agree (we normally use statistics to determine this, but use your best judgment) and put this average in your conclusion.

Questions:

1. List all the species present at the endpoint of the titrations in Part A.

2. Describe the main difference between experiments that have a limiting reactants and titrations.

READ all of the following

Report:

Complete a formal typed lab report as described in the beginning of this lab manual. You must include all sections for credit. Remember to keep the objective (Purpose) in mind when writing the report. The objective is what you want to prove so you must show all calculations and data necessary to prove it.

Everything should be written in your own words and you must turn in original work. Do NOT hand in the same report as anyone else in the lab, you will receive a zero if you are caught plagiarizing in any way.

You must include your unknown number in your lab report. Staple the following rubric to the back of your report.

Be sure to include all of your calculations for the standardization of the NaOH AND for solving for the molar mass of the unknown acid in the REPORT. Both chemical equations should be included in the report to support the mol:mol ratios you use in your stoichiometry calculations.

Your Data section should include all of the raw measurements from lab such as the masses of each sample and initial and final burette readings to 2 decimal places.

RUBRIC FOR ASSESSING MOLAR MASS OF AN ACID LAB REPORTS

	0	0.5	1	➢ 1	Score
Pre-lab	No pre-lab submitted or late	A few questions wrong but assignment is neat, or All correct but messy, or did not write the questions	Complete and neat, questions included, up to ~1 question can be incorrect.	Optional pre-lab bonus: 1.5 points if neat and perfect	
Cover Sheet	It is expected that you have a cover sheet that is complete and accurate. -0.5 for missing cover sheet.				
Objective/ Purpose	Missing section or section present but incomplete (does not mention molar mass determination), incorrect or not logical.	Complete, correct and logical			
Experimental procedure	Missing section or manual cited but incomplete or missing procedure modifications	Complete and correct			
Observations and Data	Raw data or observations missing.	Data present but no observations reported, not reported clearly in table	All raw data and observations complete, correct and in table format.		
Calculations and Results	No calculations included in the report. Results table missing.	Calculations included in the report or are in the lab notebook only, or calculations incorrect	1-2.5 points Some calculations are shown and are correct. "Equation editor" in Word, not used (-0.5). Results table incomplete/inaccurate.	3 points All calculations are shown and are correct. Results table present and accurate.	
Conclusions	Conclusions missing or missing the important points or illogical.	Conclusions regarding major points are drawn, but many are misstated, indicating a lack of understanding. Sources of error inadequate.	All important conclusions have been drawn, student shows good understanding, molar mass is reported		
Questions	No correct answers	One question answered correctly.	Both questions answered correctly.		
Accuracy	Molar mass is > +/- 10 g/mol from correct value Unknown number not included.		1 point - Molar mass is > +/- 5 g/mol and < +/- 10 g/mol from correct value	2 points - Molar mass is < +/- 5 g/mol from correct value	

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Pre-Laboratory Assignment for: Net Ionic Equations Lab

1. Vocabulary: (Please define or describe the following)
    a. Net Ionic Equation –
    b. Oxidation/Reduction Reaction –
    c. Acid/Base Reaction –
    d. Gas Forming Reaction -
    e. Single Replacement Reaction –
    f. Precipitation Reaction -

2. Consider lead nitrate and sodium chloride
    a. Write the formulas for both
    b. Would you expect lead nitrate to react with sodium chloride?
    c. Why or why not?
    d. Write the NIE.
3. For each of the reactions in the report form, complete the overall equation and identify the type of reaction it is (i.e. precipitation, gas-forming etc.). Be sure it is balanced and includes states (s), (l), (g) or (aq). Write these equations and types of reactions in the report form in pencil and be prepared to show your instructor. Feel free to also complete the total ionic equations and net ionic equations, if you have learned them in lecture, to have less to complete during the lab itself.

Net Ionic Equations Lab

Objective:
This laboratory will lead you to perform a large number of short experiments. The observation you make for each experiment will lead you to conclusions about what reactions are occurring as well as giving you the opportunity to create some generalized rules about reactivity.

Procedure:
Most of the reactions performed in this experiment will be done using small amounts in a "well plate." The well plate has 12 indentations into which you can add small amounts of reactants either as solutions or as solids. Solutions are 0.1M except sodium carbonate and sodium bicarbonate are 0.5M and hydrochloric acid and sodium hydroxide are 1.0M.

General rules for performing these reactions:

1. For reactions between two solutions, add 8-10 drops of one solution then 8-10 drops of the other solution to a well. If nothing happens, stir the solutions using a toothpick or glass stirring rod.
2. For reactions between a solid and a solution, add a **small** spatula-tip full of the solid to the well and then add 5-6 drops of the solution on top of it. Stir. If it appears that the solid might be dissolving but not completely, go ahead and add more of the solution to see if you can get it to dissolve.
3. Place a sheet of blank white paper under the well plate. Some precipitates are hard to see. Add a few more drops of the reactants to make sure you see a reaction if there is one.
4. For each mixture, you should record what you see happen as observations. In particular, does a solid form or dissolve? Does the color change? Is a gas formed? If so, what color if any? Not all of the compounds will react with each other – sometimes nothing will happen. Then again, sometimes something does happen but you can't detect it by the eye. All you can do is record what you see and go from there. It will become more apparent what happens when writing the Net Ionic equations.

NOTE: The chromate ion, CrO_4^{2-}, is not on the solubility table, it follows the same rules as the carbonate ion CO_3^{2-}.

You will NOT have a separate write-up for this lab. You will be handing in the following pages with the observations and equations as a lab report. Given the reactants, perform either a double or single replacement reaction and predict the products. Use your solubility table and knowledge of gas forming reactions to determine the states of the predicted products. Determine if there was "No Reaction" from your NIE rules not from your observations. Show the total ionic equation for all reactions. If all species, on both sides of the equation are spectator ions then write "No Reaction" for the net ionic equation and the "reaction type." Write the equations for those that you predicted reacted even if you didn't observe it. For each mixture that does react in some way, write the reaction type, overall equation, total ionic equation and the net ionic equation. All need to be balanced with the states for each compound, ion, and/or element in the reaction indicated.

Net Ionic Equations Report Form

Name_____
Lab Partner _____

Prelab (1 pt) _____

Accepted 5 pts / Accepted 3 pts / Rejected 0 pts

1. $Ca_3(PO_4)_{2(s)} + H_2SO_{4(aq)}$ Reaction Type_____

Observations	
Overall Equation	
Total Ionic Equation	
Net Ionic Equation	

2. $Ca_3(PO_4)_{2(s)} + HCl_{(aq)}$ Reaction Type_____

Observations	
Overall Equation	
Total Ionic Equation	
Net Ionic Equation	

3. $Ni(NO_3)_{2(aq)} + Na_3PO_{4(aq)}$ Reaction Type_____

Observations	
Overall Equation	
Total Ionic Equation	
Net Ionic Equation	

4. $Cu_{(s)} + AgNO_{3(aq)}$ (Form copper (II) nitrate) Reaction Type_____

Observations	
Overall Equation	
Total Ionic Equation	
Net Ionic Equation	

5. $CaCO_{3(s)} + HCl_{(aq)}$ Reaction Type_____

Observations	
Overall Equation	
Total Ionic Equation	
Net Ionic Equation	

6. $Zn_{(s)} + HCl_{(aq)}$ Reaction Type_____

Observations	
Overall Equation	
Total Ionic Equation	
Net Ionic Equation	

7. $NaHCO_{3\,(aq)} + HCl_{(aq)}$ Reaction Type_____

Observations	
Overall Equation	
Total Ionic Equation	
Net Ionic Equation	

8. $Pb(NO_3)_{2(aq)} + KI_{(aq)}$ Reaction Type_____

Observations	
Overall Equation	
Total Ionic Equation	
Net Ionic Equation	

9. $Pb(NO_3)_{2(aq)} + K_2CrO_{4(aq)}$ Reaction Type_____

Observations	
Overall Equation	
Total Ionic Equation	
Net Ionic Equation	

10. $Ca(NO_3)_{2(aq)} + Na_3PO_{4(aq)}$ Reaction Type_____

Observations	
Overall Equation	
Total Ionic Equation	
Net Ionic Equation	

11. $Fe(NO_3)_{3(aq)} + NaOH_{(aq)}$ Reaction Type_____

Observations	
Overall Equation	
Total Ionic Equation	
Net Ionic Equation	

12. $KNO_{3(aq)} + NaOH_{(aq)}$ Reaction Type_____

Observations	
Overall Equation	
Total Ionic Equation	
Net Ionic Equation	

13. $Na_2CO_{3(aq)} + HCl_{(aq)}$ Reaction Type_____

Observations	
Overall Equation	
Total Ionic Equation	
Net Ionic Equation	

14. $Ni(NO_3)_{2(aq)} + NaOH_{(aq)}$ Reaction Type_____

Observations	
Overall Equation	
Total Ionic Equation	
Net Ionic Equation	

15. $Ni(NO_3)_{2(aq)} + Na_2SO_{4(aq)}$ Reaction Type_____

Observations	
Overall Equation	
Total Ionic Equation	
Net Ionic Equation	

16. $Ba(NO_3)_{2(aq)} + Na_2SO_{4(aq)}$ Reaction Type_____

Observations	
Overall Equation	
Total Ionic Equation	
Net Ionic Equation	

Solubility Rules

SOLUBLE COMPOUNDS	EXCEPTIONS
Almost all salts of Na^+, K^+, NH_4^+	
Salts of nitrate, NO_3^-; chlorate, ClO_3^-; perchlorate, ClO_4^-; acetate, $CH_3CO_2^-$	
Almost all salts of Cl^-, Br^-, I^-	Halides of Ag^+, Hg_2^{2+}, Pb^{2+}
Compounds containing F^-	Fluorides of Mg^{2+}, Ca^{2+}, Sr^{2+}, Ba^{2+}, Pb^{2+}
Salts of sulfate, SO_4^{2-}	Sulfates of Ca^{2+}, Sr^{2+}, Ba^{2+}, Pb^{2+}, Ag^+

INSOLUBLE COMPOUNDS	EXCEPTIONS
Most salts of carbonate, CO_3^{2-}; phosphate, PO_4^{3-}; oxalate, $C_2O_4^{2-}$; chromate, CrO_4^{2-}	Salts of NH_4^+ and the alkali metal cations Na^+, K^+ are exceptions for all of these
Most metal sulfides, S^{2-}	BaS is soluble
Most metal hydroxides OH^- and oxides O^{2-}	$Ba(OH)_2$ is soluble

all acids are soluble

Strong Acids	Strong Bases
HCl	LiOH
HBr	NaOH
HI	KOH
HNO_3	$Ca(OH)_{2\,(s)}$
$HClO_4$	$Ba(OH)_{2\,(s)}$
H_2SO_4	

Weak Acids	Weak Bases
CH_3COOH	CH_3COO^-
NH_4^+	NH_3
H_2CO_3	CO_3^{2-}
$H_2C_2O_4$	$C_2O_4^{2-}$
H_2SO_3	SO_3^{2-}
H_2S	S^{2-}
H_3PO_4	PO_4^{3-}
HCN	CN^-
HF	F^-
NHO_2	NO_2^-
HClO	ClO^-

Gas Forming Reactions:

$2\,H^+ + CO_3^{2-} \rightarrow H_2O(l) + CO_2(g)$

$2\,H^+ + S^{2-} \rightarrow H_2S(g)$

$2\,H^+ + SO_3^{2-} \rightarrow H_2O(l) + SO_2(g)$

$2\,H^+ + M \rightarrow M^{2+} + H_2(g)$

M = a metal atom

Strong Electrolytes:
Soluble ionic compounds
Strong acids and strong bases

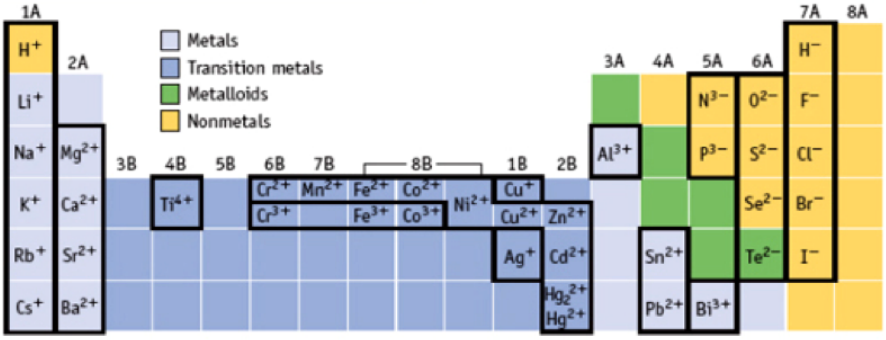


Table 3.1 Formulas and Names of Some Common Polyatomic Ions

Formula	Name	Formula	Name
CATION: Positive Ion			
NH_4^+	ammonium ion		
ANIONS: Negative Ions			
Based on a Group 4A element		**Based on a Group 7A element**	
CN^-	cyanide ion	ClO^-	hypochlorite ion
$CH_3CO_2^-$	acetate ion	ClO_2^-	chlorite ion
CO_3^{2-}	carbonate ion	ClO_3^-	chlorate ion
HCO_3^-	hydrogen carbonate ion (or bicarbonate ion)	ClO_4^-	perchlorate ion
Based on a Group 5A element		**Based on a transition metal**	
NO_2^-	nitrite ion	CrO_4^{2-}	chromate ion
NO_3^-	nitrate ion	$Cr_2O_7^{2-}$	dichromate ion
PO_4^{3-}	phosphate ion	MnO_4^-	permanganate ion
HPO_4^{2-}	hydrogen phosphate ion		
$H_2PO_4^-$	dihydrogen phosphate ion		
Based on a Group 6A element			
OH^-	hydroxide ion		
SO_3^{2-}	sulfite ion		
SO_4^{2-}	sulfate ion		
HSO_4^-	hydrogen sulfate ion (or bisulfate ion)		

Determining Net Ionic Equations

1. Write out all reactants as they exist in solution
2. Identify acids and bases
   - 2a. If both an acid and a base are present, an acid-base reaction occurs
   - 2b. Be sure to look for hidden bases that are anions in other ionic compounds, such as CO_3^{2-} in $CaCO_3$.
3. Look for ions that will form an insoluble compound. If so, they form a precipitate.
4. Look for one of the known gas-forming reactions.
5. Write out products as they exist in solution.
6. Cancel spectator ions. Note: ions that are "always soluble" will be spectator ions in acid-base or precipitation reactions.

Pre-Laboratory Assignment for: Thermochemistry: Enthalpy of Formation and Dissolution

1. Vocabulary: (Please define or describe the following)
    a. Hess's Law –
    b. Calorimetry –
    c. Thermal Equilibrium –
    d. Heat of formation –
    e. Enthalpy of Dissolution –
    f. Exothermic –
    g. Endothermic –
2. Read the lab. In your own words and in complete sentences:
    c. Describe the purpose of the lab. Why are we doing it?
    d. What concepts and/or calculations will we be using?
3. In a table, list the glassware and equipment we will be using and their purpose/function.

Glassware/Equipment	Purpose/Function
1.	
2.	

4. In a table, list the chemicals and other potentially hazardous equipment/procedures we will be using in THIS lab and how you are going to minimize your risks.

CHEMICAL/EQUIPMENT/PROCEDURE	HAZARD AND MY STEPS TO STAY SAFE
1.	

5. Part I uses three equations that will be added together using Hess's Law. Some reactants of one reaction cancel identical products in another reaction such that the net reaction is that for the formation of magnesium oxide: $Mg_{(s)} + ½\ O_{2(g)} \rightarrow MgO_{(s)}$

    Write the three equations so they cancel correctly. You may need to reverse one or more reactions or multiply by a factor. See Hess's Law in your text if this is foreign to you.

Thermochemistry: Enthalpy of Formation and Dissolution

Objective:

The purpose of Parts Ia and Ib of this experiment is to determine the enthalpy of formation of a compound, magnesium oxide, MgO. The purpose of Part II of this experiment is to calculate the heat of solution of three compounds and compare the ionic bond strength to the water-ion hydration energy.

Introduction:

The formation reaction for Magnesium oxide is:

$Mg_{(s)} + ½\ O_{2(g)} \rightarrow MgO_{(s)}$ $\Delta H_f = ?$

To determine this, we will perform two reactions and measure their enthalpy change using calorimetry.

$Mg_{(s)} + 2H^+_{(aq)} \rightarrow Mg^{2+}_{(aq)} + H_{2(g)}$ ΔH_1 = we will find this experimentally
$MgO_{(s)} + 2H^+_{(aq)} \rightarrow Mg^{2+}_{(aq)} + H_2O_{(l)}$ ΔH_2 = we will find this experimentally
$½\ O_{2(g)} + H_{2(g)} \rightarrow H_2O_{(l)}$ ΔH_3 = this can be found in the data table

Once the three values, ΔH_1, ΔH_2 and ΔH_3, are obtained, Hess's Law can be used to combine those values such that the desired heat of formation of MgO can be calculated.

Procedure:

Put the first 2 data tables in the report form into your duplicating notebook to record the raw data. Never record data into your lab manual.

PART 1a - Determining ΔH_1 and the Relation between the Quantity of Material Reacting and the Heat Transferred

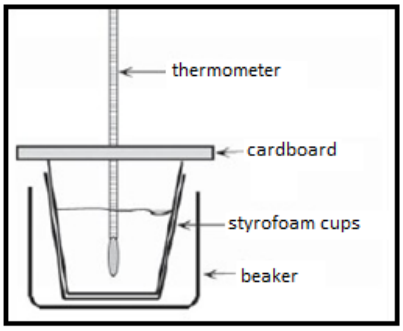
In this portion of the experiment we want to explore the relationship between the quantity of magnesium metal reacting with hydrochloric acid and the heat evolved by the reaction.

$Mg_{(s)} + 2HCl_{(aq)} \rightarrow MgCl_{2(aq)} + H_{2(g)}$

1. Set up your coffee-cup calorimeter and support it in a ring stand or beaker as illustrated. A temperature probe will be used instead of a thermometer.

(a) Calorimeter consists of a thermometer, two styrofoam cups, paper cover, and beaker.

(b) Two cups together, supported by a beaker. Cover in place with a thermometer through the hole in the cover.



2. Weigh out, to the nearest 0.001 g, three different portions of magnesium metal, about 0.2 g, about 0.4 g, and about 0.5 g. Record the masses in your lab notebook.
3. Using a graduated cylinder, measure out as accurately as possible 100.mL of 1.0 M HCl. Pour it into in a clean, dry coffee-cup calorimeter. Measure the mass and temperature of the HCl solution and record them in your lab notebook.
4. Drop one of the magnesium samples into the acid and swirl gently but steadily.
5. Stir with the probe and record the highest temperature obtained.
6. Repeat the steps above with another sample of Mg. Do all three samples, making sure the calorimeter is clean and dry each time.
7. In each case, determine ΔT, the change in temperature between the temperature of the HCl solution before adding it to the magnesium and the maximum temperature of the reacting system.

PART 1b - Determining ΔH_2: Heat of Reaction of Magnesium Oxide with Hydrochloric Acid

1. Set up the calorimeter as in PART Ia, using a temperature probe. Make sure the calorimeter is clean and dry.
2. Weigh out about 0.7 g of magnesium oxide, MgO, to the nearest 0.001 g.
3. Using a graduated cylinder, measure out as accurately as possible 100.mL of 1.0 M HCl. Pour it into in a clean, dry coffee-cup calorimeter. Measure the mass and temperature of the HCl solution and record in your duplicating lab notebook.
4. Pour the MgO powder in the calorimeter and swirl gently but steadily.
5. Repeat twice with another sample of MgO for a total of 3 trials. Do all three samples, making sure the calorimeter is clean and dry each time.
6. In each case, determine ΔT, the change in temperature between the temperature of the HCl solution before adding it to the magnesium oxide and the maximum temperature of the reacting system.

Part 1 Data table

	Mass solid (g)	Mass HCl (g)	Initial Temp (°C)	Final Temp (°C)	Change in Temp $T_f - T_i$ (°C)
Mg					
Mg					
Mg					
MgO					
MgO					
MgO					

Don't forget to record observations too.

Calculations for Parts 1a and 1b:

In each reaction, we will assume that the calorimeter absorbs no heat. Therefore, the amount of heat energy released is determined by the temperature rise of the solution. Each solution is dilute, so they are composed mostly of water and we will assume that each has a specific heat capacity of 4.18 J/g·°C.

1. Use the equation, $\mathbf{\Delta H_{rxn} + mC\Delta T = 0}$. Assume the specific heat of the solution is 4.18 J/g·°C and the mass is the sum of the acid and the other reactant. The sample calculation shown in part B is very similar and can be followed for Part A as well.

   Solve for ΔH_{rxn} in Joules. Convert Joules to kilojoules. Convert grams of the solid to moles. Divide kJ by mol to determine the value of ΔH for each reaction **in kJ/mol**.

   $$\Delta H = \frac{\#kJ}{\#\ moles}$$

   Remember that if heat is released, then ΔH must be a negative value.

2. Report the value of ΔH for each reaction.
3. Use the three reactions performed in Part 1 to verify if the amount of heat released is proportional to the number of moles of compound reacting. Report on whether this is true.
4. Use the ΔH values determined experimentally as well as that for formation of H_2O liquid to calculate the value of ΔH of formation for $MgO_{(s)}$. To do so, you must find a way of combining the three reactions for which you know ΔH so that they add up to give the reaction of interest. Remember that you can reverse reactions (and then must switch the sign of ΔH) or multiply reactions by a constant (and then must multiply the ΔH by that same constant). **Report how you do this calculation and the final value of the heat of formation of $MgO_{(s)}$ in your report.**

Part II - Exploring the heat of dissolution of select ionic compounds.

Introduction:

Dissolution of all ionic compounds involve the same basic two steps: the dissociation (or breaking apart) of the ions in the ionic crystal lattice as ions are separated and the hydration of each ion as it is surrounded by several water molecules. An example dissolution equation for sodium chloride is shown below.

$$NaCl_{(s)} \rightarrow NaCl_{(aq)} \qquad \Delta H_{diss} = 3.9 \text{ kJ/mol}$$

Enthalpy change for the conversion from a solid to an aqueous is represented by ΔH_{diss}. Dissolution of some salts is exothermic, which that of other salts can be endothermic. A negative ΔH_{diss} indicates an exothermic change, thermal energy is therefore given off by the dissolution and the resulting solution will be warmer than the initial water. A positive ΔH_{diss} indicates an endothermic change, thermal energy is therefore absorbed by the dissolution and the resulting solution will be cooler than that of the initial water. The dissolution of sodium chloride is endothermic, and requires 3.9 kJ of thermal energy to dissolve 1 mol. This much energy (q) would be removed from the surroundings (the water) and the temperature would decrease. The temperature change could be predicted using $q=mC\Delta T$.

Procedure:

1. Determine from your instructor which 2 (of the 3) compounds your group should analyze.
2. Create a data table like the one below in your NB. Make the table in "landscape" and use an entire page so you have room to make it neat, even if you have to cross out data and re-write.
3. Tare a clean dry empty Styrofoam cup.
4. Add approximately 50 mL of purified water from a graduated cylinder and record the mass of the water. Place the cup into a second cup for additional insulation.
5. Use the method of weighing by difference to measure the mass the first compound in the table below.
    a. Tare an empty balance
    b. Place a weigh boat on the pan and make a mental note of the mass.
    c. Add the solid compound to the boat until you have added the approximate required mass.
    d. Record this mass in your data table as "weigh boat + solid"
6. Place a temperature probe in the cup, cover and record the initial temperature.
7. Add the solid to the water and stir with the temperature probe but take care not to poke a hole in the fragile cup. Do not attempt to rinse any residual solid from the weigh boat. Leave it dirty but do not discard!!!
8. Watch the temperature and record the highest or lowest temperature reached.
9. Tare an empty balance and place the used weigh boat on the pan.
10. Record this mass in your data table.
11. As always record your observations.
12. Repeat the procedure for a total of 2 trials for 2 of the 3 compounds.

Part 2 Data Table: (Copy this table into your duplicating notebook in landscape format – turn your notebook sideways)

compound and trial	approx. mass of compound	mass of water (g)	mass of weigh boat and compound (g)	mass dirty weigh boat (g)	actual mass of compound used (g) (Subtract weigh boat)	initial temp of water °C	final temp of solution °C	ΔT final-initial temp
NaOH Trial 1	1 g							
NaOH Trial 2	1.5 g							
NH_4Cl Trial 1	2 g							
NH_4Cl Trial 2	2.5 g							
$Na_2SO_4 \cdot 10H_2O$	2 g							
$Na_2SO_4 \cdot 10H_2O$	2.5 g							

Don't forget to record observations too.

Calculations for Part 2:

In each reaction, we will assume that the calorimeter absorbs no heat. Therefore, the amount of heat energy released is determined by the temperature change of the solution. Each solution is dilute, so they are composed mostly of water and we will assume that each has a specific heat capacity of 4.18 J/g·°C.

1. Calculate the enthalpy change (ΔH) for each trial of each reaction/dissolution in kJ/mol of salt.
2. Average the trials that agree for each reaction and report the average enthalpy changes (ΔH) you determined in your conclusion.

The heat of dissolution is equal to the heat absorbed or released by/to the solution but with the opposite sign, therefore:

$$\Delta H_{diss} + q \text{ sol'n} = 0$$

Solve for the q the solution absorbed or released for the dissolution using $q = mC\Delta T$ where the mass is the total mass of both the water and the dissolved compound.

Sample Calculation from Sample Data:

compound /trial	approx. mass of compound	mass of water (g)	mass of weigh boat and compound (g)	mass used weigh boat (g)	actual mass of compound used (g) (Subtract weigh boat)	initial temp of water °C	final temp of solution °C	ΔT final-initial temp
KBr Trial 1	1 g	49.230	2.983	1.167	1.816	20.1	18.5	-1.6

$\Delta H_{diss} + mC\Delta T = 0$ Assume the specific heat of the solution is 4.18 J/°C * g

ΔH_{diss} + (mass water + mass KBr)(4.18 J/°C * g)(Temp final – Temp initial) = 0

ΔH_{diss} + (49.230 g + 1.816 g)(4.18 J/°C * g)(18.5°C – 20.1°C) = 0

ΔH_{diss} + (-341.722 J) = 0

ΔH_{diss} = 341.722 J

However, this needs to be reported in kJ/mol KBr. Recall this was the energy absorbed when 1.816g KBr was dissolved. Therefore, the ΔH can be written: $\Delta H = \frac{341.722\ J}{1.816g\ KBr}$

$$\Delta H = \frac{341.722\ J}{1.816g\ KBr} * \frac{1\ kJ}{1000J} * \frac{119.002\ g\ KBr}{1\ mol} = +22.39\ kJ/mol$$

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Name_____ Lab Partner's Name_____

Thermochemistry Report Form

Complete the report form neatly and in complete sentences.

Prelab _____

Accepted 5pts / Accepted 3pts / Rejected 0pts

Purpose and Procedure

(Describe the purpose **and** a summary of the experimental procedure in your own words)

Data

Part 1

	Mass solid (g)	Mass HCl (g)	Initial Temp (°C)	Final Temp (°C)	Change in Temp $T_f - T_i$ (°C)
Mg					
Mg					
Mg					
MgO					
MgO					
MgO					

Observations:

Part 2

compound and trial	Mass water (g)	mass boat and cmpd (g)	mass used boat (g)	mass cmpd (g)	initial temp °C	final temp °C	ΔT
NaOH Trial 1							
NaOH Trial 2							
NH_4Cl Trial 1							
NH_4Cl Trial 2							
$Na_2SO_4 \cdot 10H_2O$							
$Na_2SO_4 \cdot 10H_2O$							

Observations:

Calculations

Part 1a: **Show the** calculation: ΔH_1 for 0.2g $Mg_{(s)} + 2H^+$

Calculate ΔH_1 for 0.4g $Mg_{(s)} + 2H^+$

Calculate ΔH_1 for 0.5g $Mg_{(s)} + 2H^+$

Calculate Average ΔH_1 _____ J/mol_____kJ/mol

Part 1b: Calculate ΔH_2 for Trial 1 $MgO_{(s)} + 2H^+$

Calculate ΔH_2 for Trial 3 $MgO_{(s)} + 2H^+$

Calculate ΔH_2 for Trial 3 $MgO_{(s)} + 2H^+$

Calculate Average ΔH_2 _____J/mol_____kJ/mol

Hess' Law Calculation:

Enter your results into the table below, flip the equations if necessary to use Hess' Law to solve for the net reaction for the heat of formation of $MgO_{(s)}$. If you flip an equation, remember to change the sign of the heat of reaction before you add.

Enter all ΔH values in kJ/mol

Reaction 1a		ΔH_1 =
Reaction 1b		ΔH_2 =
	½ $O_{2(g)}$ + $H_{2(g)}$ → $H_2O_{(l)}$	ΔH_3 = -285.5 kJ/mol
Net Reaction		ΔH_f(MgO) =

Part 2 Calculations:

Show the calculation of ΔH_{diss} in kJ/mol for both trials for the 2 compounds you chose, and put the results for all trials and the average in the results table.

NaOH

NH_4Cl

$Na_2SO_4 \cdot 10H_2O$

Part 2 Results:

Compound	Trial 1 ΔH kJ/mol	Trial 2 ΔH kJ/mol	Average ΔH kJ/mol	Exothermic or Endothermic?
NaOH				
NH_4Cl				
$Na_2SO_4 \cdot 10H_2O$				

Question:

1. Dissolving ionic compounds involves two processes:
   1. Separating the cations from the anions. This requires input of energy.
   2. Hydrating the ions. This involves water molecules forming ion-dipole attractions with the ions. This releases energy.
   For each ionic compound you chose, use your experimental results for $\Delta H_{solution}$ to decide which is the larger effect: separating the ions or hydrating the ions. Explain each using your numerical data.

Describe at least 3 sources of error. Include how the error would affect the final result.

Pre-Laboratory Assignment for: Spectrophotometric Analysis of Copper

1. Vocabulary: (Please define or describe the following)
    a. Spectrophotometry -
    b. Beer's Law –
    c. Cuvette –
    d. Percent by mass of an element in a compound –

2. Summarize the overall purpose and procedures you will perform in this lab in your own words and explain their purpose. Write in complete sentences. Do not list the steps!
3. What is the mass percent of copper in each of the four compounds you will be using in this lab? Show your work!

4. Write the molecular equations for the double replacement reactions of each of the four compounds with nitric acid (ignore the waters of hydration since one of the reactants is an aqueous solution). If the compound is soluble in water, indicate the state as (aq). Write "no reaction" if you expect no reaction.

5. Write the molecular equations for the reactions of each of the four compounds with barium nitrate (ignore the waters of hydration since one of the reactants is an aqueous solution). If the compound is soluble in water, indicate the state as (aq). Write "no reaction" if you expect no reaction.

6. What 2 variables will you use to make your standard plot? Which will be on the y-axis?

7. Make a table of the hazards in this experiment and what are you going to do to increase your safety. Be specific to THIS experiment. For example:

HAZARD	MY STEPS TO STAY SAFE
1.	
2.	

Spectrophotometric Analysis of Copper Lab

Objective:

The purpose of this experiment is to accurately identify four unique unknown solid copper compounds. To do this you will:

•calculate and prepare standard solutions of known concentration

explore the technique of spectrophotometry, the use of light to determine some property of a substance.

• determine the weight percent of copper in each unknown compound using spectrophotometry

• observe the reactions of the four copper compounds with nitric acid and barium nitrate

You will be provided with a solid sample of four copper containing compounds. They are $CuSO_4 \cdot 5H_2O$, $CuCl_2 \cdot 2H_2O$, $CuCO_3$, $CuBr_2$ but they will not be labeled.

The concentration of copper in each of these compounds is unique and will allow you to identify 2 of the 4 compounds. The percent of the other 2 are too close to each other to be sure of their identity. You will need to use the reactions in the next part to distinguish between the last 2 compounds. Since you know the formulas for the 4 possible unknowns, you know the mass percent of copper in each. See your pre-lab. These are very important, as you will compare the percent copper of your unknowns to these to identify 2 of the compounds in Part A.

In order to determine the concentration of copper in the unknowns, you will need to construct a standard plot of the concentration of copper vs. the absorbance of solutions having known copper concentrations. According to Beer's Law, absorbance is directly proportional to concentration and so the resulting plot should be linear (a straight line.) This graph will be used to determine the concentrations of solutions containing the unknown copper compounds. From this concentration you can determine the weight percent of copper in the unknowns.

In Part B, you will combine each of the four unknowns with nitric acid and barium nitrate. With these observations, you will confirm the identity of the 2 compounds determined in Part A using percent copper as well as determine the identity of the remaining 2 compounds that had similar percent copper in their formulas.

EXPERIMENTAL PROCEDURES

Part A. Standard Plot

To determine the amount of copper in an unknown solution, you first have to explore the relationship between the concentration of copper ion and the amount of light absorbed by the sample. The important relationship is that the absorbance (A) of the solution is proportional to the concentration of the solution, c. That is, $A = \varepsilon \bullet b \bullet c$, where ε is the molar extinction coefficient (also called an absorptivity coefficient), a fundamental property of the molecules involved, and b is the length of the sample cell. In this portion of the experiment you will confirm that relation and find out what the exact relationship is for your particular spectrophotometer.

Preparing Standard Solutions of Known Copper Concentration:

1. Note: You will NOT use a graduated cylinder at all during this lab.
2. Clean and dry the large test tubes from your bin and label them Std 2, Std 3, Sample A, B, C and D.
3. Determine the mass of copper (II) nitrate trihydrate needed to make a 0.15 M solution in a 50.0 mL volumetric flask.
4. In a plastic weigh boat, measure the copper (II) nitrate trihydrate needed and record the actual mass used in your duplicating NB. Do not attempt to measure the exact amount – just get close.
5. Dissolve the crystals in a small amount of 1.0 M HNO_3 (in the weigh boat) and quantitatively transfer to the flask using a funnel. Rinse the boat and the funnel with 1.0 M HNO_3 solution. Dilute to the line on the flask with 1.0 M HNO_3. Stopper and invert many times to ensure it is fully dissolved and mixed. Once fully mixed, transfer the entire solution to a clean 100 mL beaker labeled "Standard #4."
6. Obtain 6 clean cuvettes and covers labeled 1, 2, 3, 4, A, B, C, and D. The label cannot interfere with the path of the light beam so labeling the covers is best.

   When filing the cuvettes, they should be filled ¾ full of the proper solution (see below for which solution to put in each). If the cuvettes are wet, fill with desired solution, empty and fill again. This will ensure the solution is not being diluted in the cuvette.

7. Fill cuvette #1 with 1.0 M HNO_3
8. Fill cuvette #4 from the beaker of solution you just prepared.
9. Obtain a clean 10 mL volumetric flask. Rinse it with a small amount of 1.0 M HNO_3 and discard the rinse. Pipet 4.0 mL of your stock solution ("Standard #4") into the volumetric flask. Fill to about ¾ full with 1.0 M HNO_3. Stopper and invert several times to mix thoroughly. Fill to the line using a dropper. Invert a few more times to mix.

10. Fill cuvette Std #2 with the solution and pour the remainder into the labeled #2 test-tube.
11. Re-rinse the 10mL volumetric flask with 1.0 M HNO_3. Repeat the same procedure for standard #3, this time pipetting 7.0 mL of standard #4 into the volumetric flask.
12. Fill cuvette #3 with the solution and pour the remainder into the labeled #3 test-tube.

Copy the following table in your notebook under "Data":

Standard #	Volume HNO_3	Volume Cu^{2+} Standard	Molarity of Cu^{2+}	Absorbance
1	Fill cuvette (¾ full)	0 mL	0	0.00
2	6.0 mL	4.0 mL		
3	3.0 mL	7.0 mL		
4	0 mL	Fill cuvette (¾ full)		

Preparing Unknown Solutions:

Obtain 4 copper samples from your instructor. Record the color of each in the table below. Copy the following table in your duplicating notebook under "Data."

Unknown	Color	Mass (g)	Absorbance
A			
B			
C			
D			

1. Re-rinse your 10 mL volumetric flask with 1.0M HNO_3. Measure approximately 0.15g of unknown A in a weigh boat and record the exact mass in your duplicating notebook (record all decimal places).
2. In the weigh-boat, dissolve the solid in a small amount of 1.0 M HNO_3 and transfer to the 10mL volumetric flask. Rinse the boat with 1.0M HNO_3 until completely transferred, but be careful not to fill above the line on the neck of the flask! Record your observations when you add the nitric acid to the solid. Enter these observations in the "reactions" table for Part B.

3. Fill the flask to the line using a dropper, stopper and invert until the solution is homogeneous.
4. Fill cuvette "A" ¾ full with the solution and cap. If the cuvette is wet, fill with desired solution, empty and fill again. This will ensure the solution is not being diluted in the cuvette.

Measuring the Absorbance

Check that the spectrophotometer's wavelength has been set to 780nm. Zero the spectrophotometer using 1.0 M nitric acid, read and record the absorbance of each solution in the data table in your notebook.

Calculations

Calculate the molarity of your Standard #4 solution using the actual mass you measured into the 50.0mL flask.

From the actual molarity of standard #4, calculate the molarity of standards #2 and #3. Because you are simply diluting a copper-containing solution of known concentration, you can use the equation: $M_1V_1=M_2V_2$ and the volume of std #4 you pipetted into the 10mL flask.

(Molarity of std #4)(mL of Cu^{2+} solution used) = (Conc. of dilute solution)(10.0 mL total volume)

Part B. Reactions

The percent mass of copper in the sample will not be enough to differentiate between all four compounds. Further data about these compounds is needed.

Copy the following table in your notebook under "Data." Make the table large enough to write a sentence or two in each box because you will record observations in the table.

Part B Data: Reaction **Observations** (Do not write "yes" or "no." Observations are what you saw, heard, smelled etc.)

Unknown	Reaction with Barium Nitrate	Reaction with Nitric Acid
A		
B		
C		
D		

1. Obtain a well plate and add a small (I mean tiny!) amount (spatula tip only) of each unknown solid to 4 separate wells.
2. Add a small amount of water to each and stir with a CLEAN glass stir rod or wooded stick. Attempt to dissolve the solid. If it does not dissolve you may have too much solid. Add a few more drops of water and/or start over with a smaller amount to be sure.
3. To the unknown/water mixture, add a few drops of barium nitrate solution and stir. Add a few more drops to be sure of your observations. Record your observations in the data table in your notebook.
4. If you did not record your observations of the unknowns with nitric acid in part A, repeat steps 1 and 2 to make fresh samples of compounds A-D. Add nitric acid to each and record your observations.
5. Check your pre-lab for the products of these reactions. Did you observe what you expected? For example, how many unknowns did you expect to form a precipicate with barium nitrate? Did you observe that many cloudy solutions? How about bubbles with the nitric acid? If you did not observe what you expected, repeat the experiments.

Waste from Part A and Part B should be collected and should not go down the drain.

Handling the Data and Performing Calculations

Use Excel to construct a scatterplot of absorbance *(vertical or y-axis)* vs. concentration *(horizontal or x-axis)* for the four standard copper solutions (cuvettes1-4). Add a linear trend-line and display the equation of the line. Title and label the axis. Print the graph and attach it to your report. Reference the "graphing practice" exercise provided in the first lab.

Calculation for % mass Cu in unknowns

Determine the mass of copper (in grams) in each of the unknown samples and use this information to determine the mass of copper in your original solid samples.

For each of the unknown samples:

1. replace the 'y' in the trend-line equation with the recorded unknown absorbance, and solve for x. This is the copper concentration of the unknown in M (moles/L). Enter this under Molarity of Cu^{2+} in the "Calculated Results" Table.
2. Multiply by 0.01 L (since the unknowns were dissolved in 10ml) and you will have the moles of copper in the original test tube.

3. Convert moles into grams of Cu.
4. Divide grams copper by grams of original sample (about 0.15 g) * 100% for the percent copper in the unknown. Record these % mass Cu results for A-D in your data table above.
5. Record the weight percent of copper for each of the possible unknowns (based on their formula) in the "Calculated Results" table. You calculated these as part of your pre-lab assignment. Compare these known weight percent values to the calculated weight percent values for your unknowns.
6. Compare the unknown % mass Cu you just calculated to the % mass copper of the 4 possible compounds and the observations in Part 2 to determine which of the possible unknown compounds are A, B, C and D. Record each identity in the same table. You must use the results of BOTH parts A and B to determine the identities of the 4 unknowns.

Copy the following tables in your report under "Calculated Results."

Calculated Results

Unknown	Color	Molarity of Cu	Moles Cu	Grams Cu	Calculated % mass Cu	Identity
A						
B						
C						
D						

(see your pre-lab answers and show the calculations in your report)	Actual % mass of Copper (calculated from the formula)
$CuSO_4 \cdot 5H_2O$	
$CuBr_2$	
$CuCO_3$	
$CuCl_2 \cdot 2H_2O$	

Report:

Type a formal written lab report as designated in the beginning of this lab manual. You must include all sections for credit. Remember to keep the objective in mind when writing the report. The objective is what you want to prove so you must show all calculations and data necessary to prove it.

Everything should be written in your own words and you must turn in original work. Do NOT hand in the same report as anyone else in the lab, you will receive a zero if you are caught plagiarizing in any way.

In your conclusion, for each of the four compounds, report the percent copper, your conclusion as to the identity of each unknown copper compound and a clear rationale as to how you came to that conclusion. Calculate and report the % error of your % mass of copper for each of the four unknowns, assuming your concluded identities are correct.

$$\%\ Error = \frac{|calculated\ \%\ Cu - actual\ \%\ Cu|}{actual\ \%\ Cu} * 100\%$$

For example: I conclude that compound A is _____ because my measured % Cu was____ and the actual % Cu for (the compound you think it is) is _____ and they are very close to each other with a % error of_____. Also compound A did not react with nitric acid and it produced a precipitate with the barium ion which is exactly how (your concluded compound) would react by the following chemical equation: XX + XX…..

I conclude that compound B is_____ …..

Attach the following rubric to the back of your report.

RUBRIC FOR ASSESSING SPEC ANALYSIS OF COPPER LAB REPORTS

	0	0.5	1	➢ 1	Score
Pre-lab	No pre-lab submitted or late	A few questions wrong but assignment is neat, or all correct but messy, or did not write the questions	Complete and neat, questions included, up to ~1 question can be incorrect.	Optional pre-lab bonus: 1.5 points if neat and perfect	
Cover Sheet	-0.5 for missing cover sheet.				
Objective/ Purpose	-0.5 for missing or illogical purpose				
Experimental procedure	-0.5 for missing or incorrect procedure or missing procedure modifications				
Observations and Data	Raw data or observations missing.	Data present but no observations reported, or not reported clearly in table, or missing some data.	All raw data and observations complete, correct and in table format.		
Calculations and Results	No calculations included in the report	Calculations included in the report or are in the lab notebook only, or calculations incorrect	Some calculations are present and correct but not all	2.0 points All calculations are present and correct.	
Beer's Law Plot	No plot shown, or hand drawn.	Plot provided but incorrect in some way.	Plot included and correct. Absorbance must be on the y axis, trend-line present (not line to connect the points), equation for the line must be present, title present and axis labeled		
Part B used in analysis	Did not use qualitative data to help identify the compounds.	Used qualitative data incorrectly.	Correctly used qualitative data from reactions to help conclude ID of compounds		

	0	0.5	1	➢ 1	Score
Conclusions	Conclusions missing or missing the important points or illogical.	Conclusions regarding major points are drawn, but many are misstated, indicating a lack of understanding.		2 points All important conclusions have been drawn, % error and **clear rationale for each identification included as described in the manual!**	
Accuracy	None of the 4 compounds are identified correctly			Up to 2 points 0.5 points for each	
Appearance and formatting	-0.5 points for grammar/spelling errors, out of order, too much handwritten copy, superscripts and subscripts not used for charges and formulas and/or poorly formatted				

Pre-Laboratory Assignment for: HANDS-ON LAB EXAM - Analysis of a Copper Complex

1. Vocabulary: (Please define or describe the following)
    a. Gravimetric analysis -
    b. Volumetric analysis –

2. What is the purpose of this experiment? Use complete sentences.

3. In a table, list the glassware and equipment we will be using and their purpose/function.

Glassware/Equipment	Purpose/Function
1.	
2.	

4. In a table, list the chemicals and other potentially hazardous equipment/procedures we will be using in THIS lab and how you are going to minimize your risks.

CHEMICAL/EQUIPMENT/PROCEDURE	HAZARD AND MY STEPS TO STAY SAFE
1.	

Notes:

Bring a lap-top if possible to lab.

Once the week of this lab begins, instructors and TA's are not allowed to answer questions so please review the lab and calculations and ask questions in advance.

EXAM - Analysis of a Copper Complex:

Introduction:

You may work in pairs to gather the data required for this exam but you must complete the calculations independently. Instructors and TA's are not allowed to help you once the week of the exam begins. Please ask questions in advance.

You will perform three separate analyses to determine the amount of Cu^{2+}, SO_4^{2-} and NH_3 in an unknown copper-containing hydrate. The quantitative analyses that you will perform are of three types: gravimetric, volumetric, and spectrophotometric. The gravimetric analysis is for sulfate ions; the volumetric analysis is for ammonia molecules; and the spectrophotometric analysis is for copper ions, all of which you performed in previous experiments. With careful attention to detail and techniques one can obtain excellent results for each part of the analysis.

You will then use your results to propose a formula for the compound. The general formula for the compound is: $Cu_x(NH_3)_y(SO_4)_z \cdot aH_2O$, where x, y, z, and a are integers which you will determine from the analysis of your product. Water content will be determined by mass difference, once the proportions of the other species are known.

As you go through this lab, there will be example calculations for each part of the lab. Use your Report Form to do your calculations. You will not do a formal written report for this lab. Instead, you will be handing in the Report Form to count as your hands-on lab exam (20pts).

Procedure:

A. Gravimetric Analysis for Sulfate Ion, SO_4^{2-}

You will determine the amount of sulfate in the copper compound by reacting a sample of the compound first with nitric acid and then lead acetate, which will cause the sulfate to precipitate as lead sulfate. You will complete two trials for this part of the lab. The two reactions involved in this process are:

$$Cu_x(NH_3)_y(SO_4)_z \cdot aH_2O_{(s)} + yH^+_{(aq)} \rightarrow xCu^{2+}_{(aq)} + yNH_4^+{}_{(aq)} + zSO_4^{2-}{}_{(aq)} + aH_2O_{(l)}$$

$$Pb^{2+}_{(aq)} + SO_4^{2-}{}_{(aq)} \rightarrow PbSO_{4(s)}$$

1. Grind the copper compound in a mortar to a fine powder. Weigh 2 samples of about 0.9-1.1 g of the ground copper compound to the nearest 0.001 g directly into 2 small beakers. Label the beakers trial 1 and 2.
2. Don gloves and a lab coat or apron if you have not done so already. Using the pump dispenser provided, carefully add 10 mL of 6 M nitric acid (HNO_3) to each beaker. Make sure the samples are completely dissolved before moving on. Crush small pieces with glass stirring rods and/or set them aside while you set up for the filtration. Use 2 stir rods – do NOT contaminate one with the other.

3. For each trial, obtain 6 mL of 1M Pb^{+2} (this may be lead acetate or lead nitrate) and add it slowly to the acid solution until precipitation is complete. Stir well.
4. Label 2 pieces of large filter paper on the edge with pencil as trial 1 and 2 and your initials. Weigh them to the nearest milligram and record the masses in your notebook. Assemble 2 apparatuses for gravity filtration. Fold the paper into a cone and place it in the funnel, moisten it with a little water, and adjust it so that it fits the funnel snugly. Be careful not to tear the paper. Carefully fill the filter cone about half full of the lead sulfate mixture. When this has drained nearly empty, repeat the operation, and continue until the transfer of lead sulfate to the filter is complete. Be careful not to lose precipitate during this transfer.
5. The filtration described above may be quite slow.
6. After most of the liquid has collected from the filtration, check it for completeness of precipitation by adding a few drops of 1 M Pb^{+2} to the solution in the flask. If a white precipitate forms (i.e., the solution becomes cloudy), add another milliliter of 1 M Pb^{+2} solution and re-filter.
7. Rinse the beaker with small portions of water and use this rinsing to wash the precipitate on the filter paper. Finally use your wash bottle to rinse the filter paper and precipitate free from the original copper-containing solution.
8. If your filter paper is blue, start over, you didn't dissolve the copper compound completely.
9. Rinse the precipitate with a few mL of acetone from the squeeze bottle (no need to measure).
10. When the liquid from the last washing has drained out of each, remove the filter paper (be careful not to tear them!) and place them in an evaporating dish to dry until next lab.

(Next Week:

This lab report will be due in 2 weeks. Next week you will obtain the dry mass of the $PbSO_4$ samples so you may complete the calculations and ultimately the entire report.

Complete this step next week on your dry samples:

1. Weigh each of your $PbSO_4$ precipitates and filter papers to the nearest milligram, record each mass in your notebook **and** on the first and second page of your report form (as "mass of $PbSO_4$"). As part of your report, calculate the mass percent in your notebook and on the report form, use the example below as a guide.)

B. Spectrophotometric Analysis for Copper, Cu^{2+}

1. Clean and dry 4 large test tubes, and label them 2, 3, A and B. Solutions 1 and 4 are made for you and can be used directly to fill the cuvette. The first four of these solutions will be used to make up the reference solutions that you will use to produce a ***standard curve***.
2. Prepare your standards and samples of the unknown compound as described in the table below using the burettes provided. Record the concentration of the standard Cu^{2+} solution. Once the solutions are prepared, fill cuvettes ¾ full and cap with labeled caps.

Tube #	Contents
1	**Fill cuvette with 1 M HNO_3**
2	**4.0 mL standard Cu^{2+} solution and 6.0 mL 1 M HNO_3. Mix well with stir rod.**
3	**7.0 mL standard Cu^{2+} solution and 3.0 mL 1 M HNO_3. Mix well with stir rod.**
4	**Fill cuvette with standard Cu^{2+} solution. Record the concentration.**
A	**Weigh ~0.2 g of ground Copper Compound to the nearest 0.001 g. Add exactly 10.0 mL 1 M HNO_3. Mix well and fill cuvette.**
B	**Weigh ~0.3 g of ground Copper Compound to the nearest 0.001 g. Add exactly 10.0 mL 1 M HNO_3. Mix well and fill cuvette.**

3. Take all of your cuvettes and your notebook to one of the spectrophotometers. Wipe the sides clean with a lint free wipe (Kimwipe).
4. Measure the absorbance of each standard and sample at 780 nm. Use the contents of tube 1 as the reference sample to zero the instrument. Record your measurements in your notebook and include an absorbance of "0.00 abs" for standard #1.

C. Volumetric Analysis for Ammonia, NH_3

The amount of ammonia in your compound will be determined by a conventional acid-base titration. The reaction that occurs during the titration is:

$NH_{3(aq)} + H^+_{(aq)} \rightarrow NH_4^+{}_{(aq)}$ where H^+ is from a standardized solution of strong acid, HCl.

1. If necessary, grind your copper compound to a very fine powder in a mortar.
2. Weigh two (approxim ples of your compound to the nearest 0.001 g and record their masses. Dissolve each in about 30mL of purified water in an Erlenmeyer flask. (Do not allow the solutions to stand very long before titrating.)
3. Add 10 drops methyl orange indicator to each solution, and titrate each with thorough stirring with standard HCl. Record the exact concentration of the HCl solution in your notebook, along with the beginning and final burette readings.
4. Check the precision of your results by dividing the volume of acid used by the mass of the sample used in each titration; if the two runs do not agree to within 1%, do a third titration.

Numerous color changes will occur as the acid is added. As acid is added, the color changes to green and then to a pea-green when about 70% of the acid has been added. After about 85% of the ammonia has been titrated, the color is distinctly yellow. The end point is a change from yellow-orange to red-orange. However, the end point detection is made easier by the fact that the precipitate that was present through the titration vanishes just before the end point. WATCH FOR THIS!

LAB EXAM - Analysis of a Copper Complex Report Form

Complete the report form neatly and in complete sentences.

Name_____ Lab Partner's Name_____

Sulfate Mass Percent Calculation

EXAMPLE OF MASS PERCENT CALCULATION for SO_4^{2-} (this is only an example, you will use your data for the masses)

Mass of copper compound used in gravimetric analysis = 1.089 g
Mass of $PbSO_4$ isolated = 1.252 g

- Calculate the moles of $PbSO_4$

$$1.252\,g\,PbSO_4 \times \frac{1\,mol\,PbSO_4}{303.25\,g\,PbSO_4} = 0.004129\,mol\,PbSO_4$$

- Use the stoichiometry from equation *(2)* to determine the moles of SO_4^{2-}

$$0.004129\,mol\,PbSO_4 \times \frac{1\,mol\,SO_4^{2-}}{1\,mol\,PbSO_4} = 0.004129\,mol\,SO_4^{2-}$$

- Convert the moles of SO_4^{2-} to grams

$$0.004129\,mol\,SO_4^{2-} \times \frac{96.06\,g\,SO_4^{2-}}{1\,mol\,SO_4^{2-}} = 0.396594\,g\,SO_4^{2-}$$

- Use the grams of SO_4^{2-} and the mass of the copper compound to determine the mass % of SO_4^{2-}

$$\frac{0.396594\,g}{1.089\,g} \times 100\% = 36.418\%$$

GRAVIMETRIC ANALYSIS FOR SULFATE CONTENT

	Sample 1	Sample 2
Mass of sample (g)		
Mass of $PbSO_4$ (g)		
Mass % of SO_4^{2-} in unknown compound		
Average mass %		

Write the equation for the precipitation reaction below:

Sample of mass % calculation: (Show the calculation for both trials below)

EXAMPLE OF MASS PERCENT CALCULATION for Cu^{2+}

Calculate the diluted concentration of Cu^{2+} in tubes 2 and 3 using the dilution equation $M_1V_1 = M_2V_2$
The final volume for all tubes is 10.0mL.

For example: $(4.0\ mL)(0.15\ M) = (10.0mL)(\ M_2)$

Show dilution calculations for standards 2 and 3 below and complete the data table.

Data table for Standard Solutions:

Tube	Concentration of Cu^{2+} in M	Absorbance
1	0.00 M	0.000
2		
3		
4		

Refer to the "graphing practice" exercise you completed during the first week of lab. Prepare a scatter plot of absorbance vs. concentration for data obtained from tubes 1-4. Add a line of best fit (trend-line) and display the equation for the line on the plot. Label the axis and title the graph. Print the plot and attach it to your report form. This plot is your "standard curve."

Write the equation for the trendline of your standard curve: _____

Use the equation for the trend-line to determine the Cu^{2+} concentration of the 2 samples, A and B.

For example, if the data were as follows:

Mass of sample A = 0.215 g

Absorbance for sample A = 0.390 abs

Equation for the trend-line: y=4.7517x + 0.0084

Find $[Cu^{2+}]$ by replacing "y" in the equation with the sample's absorbance and solving for "x" which is the concentration in molarity.

0.390abs = 4.7517x + 0.0084

x=0.080308M This is the copper ion concentration in the 10.0 mL of sample A.

List the data and show the calculations for the copper ion concentration in samples A and B below and enter in the results table.

Sample A:

Sample B:

Next you must convert this sample concentration to the mass % of copper:

Find the total moles of copper in the 10mL sample as shown in this example:

$$10.0\ mL * \frac{1L}{1000\ mL} * \frac{0.080308\ mol}{1\ L} = 8.0308\ x\ 10^{-4} mol\ Cu^{2+}$$

Convert moles to grams:

$$8.0308\ x\ 10^{-4} mol\ Cu^{2+} * \frac{63.545g}{1\ mol} = 0.051032\ g\ Cu^{2+}$$

Find mass percent copper in the synthesized compound:

$$\frac{0.051032\ g\ Cu\ in\ sample}{0.215\ g\ total\ mass\ of\ sample} * 100\% = 23.7\%$$

Show the calculations for the % copper for samples A and B below and enter in the results table.

Sample A:

Sample B:

Results table:

Sample	Mass (g)	Absorbance	Concentration of Cu^{2+} (M)	Mass % of Cu
A				
B				
			Average	

EXAMPLE OF MASS PERCENT CALCULATION for NH_3 (this is only an example, you will use your data for HCl molarity and volume)

Mass of copper compound used in titration = 1.023 g
[HCl] = 0.512 M Volume of HCl used in titration = 30.31 mL

- Calculate the moles of HCl used in titration

$$0.03031\ L\ HCl \times \frac{0.512\ mol}{1L} = 0.015519\ mol\ HCl$$

- Use the stoichiometry to determine the moles of NH_3

$$0.015519\ mol\ HCl \times \frac{1\ mol\ NH_3}{1\ mol\ HCl} = 0.015519\ mol\ NH_3$$

- Convert the moles of NH_3 to grams

$$0.015519\ mol\ NH_3 \times \frac{17.03\ g\ NH_3}{1\ mol\ NH_3} = 0.264284\ g\ NH_3$$

- Use the grams of NH_3 and the mass of the copper compound to determine the mass % of NH_3

$$\frac{0.264284\ g}{1.023\ g} \times 100\% = 25.834\%$$

VOLUMETRIC ANALYSIS FOR AMMONIA

Concentration of standardized HCl solution= _____M

	Sample 1	Sample 2	Sample 3 (if needed)
Mass of sample (g)			
Amount of HCl added (mL)			
Mass % of NH_3 in sample			
Average mass %			

Sample of mass % calculation: (Show the calculation for both trials below)

PUTTING IT ALL TOGETHER: Determining the Empirical Formula

Once the mass % of each component is determined (average the results of the separate trials), the formula for the compound can be determined.

The following table uses the data from the examples given on the previous pages.

	Weight Percent = g in 100g	Moles per 100g	Mole Ratio	
Cu^{2+}	**23.74**	0.374	0.374/0.374	*x=1*
NH_3	**25.834**			*y=*
SO_4^{2-}	**36.418**			*z=*
Total Mass of Cu^{2+}, NH_3, and SO_4^{2-}	23.74 + 25.834 + 36.418 = 85.992			
H_2O = 100 - (total of Cu^{2+}, NH_3, and SO_4^{2-})	100 - 85.992 = **14.008**			*a=*

Calculation of "Moles per 100 g" for each component

Assuming there is 100 g. If your sample is 23.74% Cu then it would contain 23.74 g Cu. Change your %'s into grams in the table below.

Using the example below, convert grams of each component to moles of each component. Show the calculations here and enter your results in the table as well.

Example conversion of grams copper to moles of copper:

$$23.74\ g * \frac{1\ mol\ Cu^{2+}}{63.545\ g} = 0.3736\ mol\ Cu^{2+}$$

Show the calculation to moles below for each of the three components analyzed:

To get the "Mole Ratio" for each component, divide by the # moles of each component by the smallest to hopefully obtain whole number ratios. In the above example, the moles for NH_3, SO_4^{2-}, and H_2O would each be divided by 0.374mol. Multiply decimals by a factor, or round to the nearest whole number. If you obtain less water than copper (or negative water) you must round up to 1 water or assume zero waters of hydration. Note: The copper should have the smallest number of moles. If it does not, check your calculations and then divide by the moles of copper regardless.

Show the division of the moles of each component by the smallest number of moles below. If needed multiply by a factor to obtain whole numbers, or if close, round to whole numbers. Complete the table below with your results.

	Mass in g	**Mol/100g**	**Mole Ratio**	**Whole # in formula**
Cu^{2+}				
NH_3				
SO_4^{2-}				
Total mass of 3 above				
H_2O (mass = 100g-total above)				

Formula of the compound: _____

Molar mass of the compound: _____

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Pre-Laboratory Assignment for: Lewis Structures and Molecular Models for Covalent Compounds Lab

1. Vocabulary: (Please define or describe the following)
    a. Lewis structure -
    b. Formal charge –
    c. Resonance structures –
    d. VSEPR theory -

2. Summarize the purpose of this lab? Use your own words and complete sentences

3. In your notebook for this prelab, draw the best Lewis structure for each of the compounds listed in the report form.
4. Copy the following table into your NB and complete the missing information. You will use this table while completing the lab.

# of Domains (electron pairs) around central atom	# of lone pairs	Electron Pair Geometry	Molecular Geometry	Bond Angles	Hybridization of central atom
2	0	Linear			
3	0			120˚	
	1		Bent		
4	0				
	1				
	2				sp^3
5	0			90˚, 120˚	
	1				
	2				
	3				
6	0				
	1				
	2				
	3				

Lewis Structures and Molecular Models for Covalent Compounds Lab

Introduction

The purpose of this lab is to gain experience drawing correct Lewis structures for molecules of covalent compounds, predicting their structures, and building models of those molecules to visualize their shapes in three dimensions. You will evaluate the geometries, polarities, resonance, and hybridization of a series of molecules. Many physical and chemical properties of compounds depend upon the shape or geometry of the molecule and its polarity. For example, molecular shape and polarity help determine a compound's boiling point, freezing point, and viscosity.

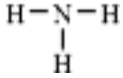
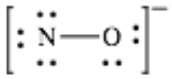
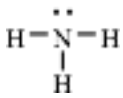
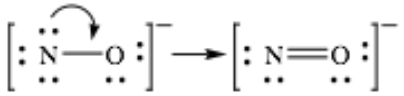
Drawing Lewis Structures: While Lewis dot structures are fairly easy to draw for simple compounds such as Cl_2 and H_2O, there are other compounds for which it is not immediately obvious how a dot structure should be drawn. If you follow the steps below, you will be able to determine the correct structure every time. This lab will help you gain experience in using these steps so you will be able to write Lewis dot structures for complex molecules and ions in this lab.

Steps to draw Lewis Dot Structures

Terms to know:

Valence electrons: Equal to group number (for the elements in the "A" groups in the periodic table).
Octet rule: Most elements are "satisfied" when sharing eight electrons. Exceptions: H (2), Be (4), B (6), Al (6)
Terminal atom: Atom attached to only one other atom in the molecule.
Central atom: Any non-terminal atom in a structure.
Bonding pair: A pair of electrons represented by a line. These electrons hold two atoms together via a chemical bond.
Nonbonding pair (lone pair): A pair of electrons that does not participate in bonding. Represented by a pair of dots associated with one atom.

The Steps:	**An example, NH_3**	**An example, NO^-**
1. Count the total number of valence electrons in the molecule. Anions: add 1 electron per charge Cations: subtract 1 electron per charge	One N = 5 Three H's = (3)(1) = 3 Total = 8 valence electrons (4 pairs)	One N = 5 One O = 6 (–1) charge = 1 Total = 5+6+1 = 12 valence electrons (6 pairs)

2. Arrange atoms (the central atom is the one farthest from fluorine on the periodic table). Occasionally you will be told how to arrange the atoms. Exception: H is NEVER a central atom	H N H H	N O
3. Add bonds between the central and terminal atoms (1 bond = 2 electrons).		N—O
4. Count the total number of electrons that you have used to make bonds. Any unused electrons are added to the terminal atoms (2 at a time, in pairs) until the octet rule is satisfied. Exception: Hydrogen NEVER has lone pairs.	Three bonds = (2)(3) = 6 electrons. Two unused electrons, but hydrogen cannot have more than two electrons. Hydrogen NEVER has lone pairs.	One bond = 2 electrons, so there are 10 unused electrons (5 pairs) (It is not possible to satisfy the octet rule for both atoms at this stage) 
5. Add any leftover electrons (in pairs) to the central atom.		Does not apply
6. Check octet rule. If an element is not satisfied, change a non-bonding electron pair (lone pair) into a bonding pair. Continue only until the octet rule is satisfied for all elements (don't forget about the exceptions).	All elements fulfill the octet rule. Final structure is shown in step 5.	Oxygen has 6 electrons and therefore needs 2 more electrons to satisfy the octet rule. Convert a lone pair on N to a bonding pair, making a **double bond** between N and O. 
7. Check formal charges if central atom can expand its octet or if multiple bonds can be in different locations.	PO_4^{-3} all single bonds fulfill the octet rule	1 or 2 double bonds reduce formal charges, but 1 double bond has (-1) on more electronegative oxygen atom.

Resonance:

Some molecules have more than one valid Lewis structure. If a molecule has two or more valid Lewis structures that differ only in the arrangement of electrons, and not the arrangement of atoms, then it has resonance. In this lab, you will draw one resonance structure when necessary and will indicate the number of valid resonance structures there are for the molecule. Resonance occurs only in compounds that can contain multiple bonds.

Formal Charge:

If more than one Lewis structure can be drawn that satisfies the octet rule, it is necessary to determine the formal charges of all the atoms to determine the best resonance structure. This is often the case when a central atom can expand its octet (third period or below) because the octet can be satisfied with single bonds but can expand to accommodate double bonds. The best resonance structure has smallest formal charges (closest to zero). If an equal spread of formal charges is possible for more than one structure, then the correct structure is the one with the negative formal charge residing on the more electronegative atom.

Molecular Polarity:

Covalent molecules can be classified as polar or nonpolar. A polar molecule contains polar covalent bonds that are asymmetrically arranged. This lack of symmetry causes an uneven distribution of electron density around the central atom that leaves part of the molecule with a negative region, and a different part of the molecule with a positive region.

Model Kits:

Molecular model kits are used to visualize the shapes of molecules. They are useful because they represent the molecules in three dimensions, as opposed to two-dimensional drawings. Chemists learn to automatically think of these 3D structures when looking at a 2D drawing. You will be building models of several molecules. First you will determine and draw the best Lewis

structure. Be sure to build a model of your molecule before answering any of the other questions especially before deciding its shape and polarity. The different colored balls represent certain atoms, according to the chart below. You can either use the tear-drop shape to represent lone pairs, or you can use balls with the holes missing in the location of the lone pairs. For example, a red oxygen ball could have 2, 3 or 4 holes, but they will always be in the tetrahedral arrangement so that the one with 2 holes will be in the "bent/109.5" arrangement and the one with 3 holes will be in the trigonal pyramidal arrangement. Beware of the central atoms that are in the third period or below (like sulfur). Since they can expand their octet they can have different electron pair geometries (4 tetrahedral, 5 trigonal bipyramidal or 6 octahedral). **Be sure to choose the correct number** of holes in the colored ball that you choose for these central atoms!

Color	Element
Black	Carbon
White	Hydrogen
Red	Oxygen
Blue	Nitrogen
Purple	Phosphorous
Green	Any Terminal Halogen
Yellow	Sulfur
Any color with the correct number of holes	Central: halogen, noble gas or other

The **short grey links** are used to represent single covalent bonds. **Two longer flexible grey links** are used to represent a double bond, and **three flexible grey links** are needed for a triple bond.

Lab Procedure

Complete the information required in the attached tables neatly. Draw the Lewis structure and make a model of each molecule and have your instructor initial under the formula before taking it apart. For any molecule or ion with resonance structures, draw one resonance structure and indicate the total number of resonance structures. Look at the model to determine the symmetry and therefore the polarity of the molecule. Be sure to

draw and construct models for all of your molecules in the time allowed. The rest of the columns can be finished outside of lab if needed.

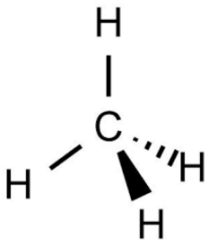
Drawing Wedge-and-Dash Structures

You now understand the connection between Lewis structures and molecular shapes and are familiar with those three-dimensional shapes. Finally, chemists need to describe three-dimensional molecular structures in two-dimensional drawings. To do this, we use drawings that imagine the molecule imbedded in a piece of paper and indicate whether each atom is split by the paper, in front of the paper, or behind the paper. That is, the drawing indicates whether an atom is in the plane of the paper, is coming towards you, or is away from you. These are called wedge and dash drawings and the bonds tell the tale of the three-dimensional shape.

A line indicates a bond that is in the plane of the paper, connecting two atoms that are in the plane of the paper.

A wedge represents a bond between two atoms where the thick side of the wedge is connected to an atom that is closer to you.

A dash represents a bond to an atom that is farther from you. In the structure here, the top H, the C and the lower left H are all in the plane of the paper. The H atom bonded with wedge is closer to you than the paper (and the C atom). The H atom bonded with the dashed line is farther from you than the paper (and the C atom).



Draw wedge and dash depictions for CH_2I_2 and for SF_6.

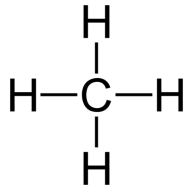
Name _____ Date_____ Pre-lab (1pt) _____

Partner _____ Accept 5pts / Accept 3pts / Reject 0pts

Report Form- Lewis Structures and Molecular Models for Covalent Compounds Lab

*If resonance, draw only 1 structure but consider the resonance when you calculate bond order.

The first row is completed for illustration.

	Best Lewis Structure* indicate non-zero formal charges next to each atom i.e. (+1)	**Electron Pair Geometry around each central atom**	**Molecular Geometry around each central atom**	**Bond Angles (BA) and Bond Orders (BO)**	**Polar or Non-Polar Molecule?**	**Hybridization around each central atom**	**Build the Molecule and get Instructor's Initials below**
CH_4	(0) For all formal charges 	C-tetrahedral	C-tetrahedral	(BA) H-C-H=109.5 (BO) C-H = 1	Non-polar	C - sp^3	VLD
PH_3							

	Best Lewis Structure* indicate non-zero formal charges next to each atom i.e. (+1)	**Electron Pair Geometry around each central atom**	**Molecular Geometry around each central atom**	**Bond Angles (BA) and Bond Orders (BO)**	**Polar or Non-Polar Molecule?**	**Hybridization around each central atom**	**Build the Molecule and get Instructor's Initials below**
H_2S							
CO_2							
NO_3^-							

	Best Lewis Structure* indicate non-zero formal charges next to each atom i.e. (+1)	**Electron Pair Geometry around each central atom**	**Molecular Geometry around each central atom**	**Bond Angles (BA) and Bond Orders (BO)**	**Polar or Non-Polar Molecule?**	**Hybridization around each central atom**	**Build the Molecule and get Instructor's Initials below**
CH_2I_2							
O_3							
CH_2O							

	Best Lewis Structure* indicate non-zero formal charges next to each atom i.e. (+1)	**Electron Pair Geometry around each central atom**	**Molecular Geometry around each central atom**	**Bond Angles (BA) and Bond Orders (BO)**	**Polar or Non-Polar Molecule?**	**Hybridization around each central atom**	**Build the Molecule and get Instructor's Initials below**
PO_4^{3-}							
SO_2Cl_2	S is central.						
SCl_4							

	Best Lewis Structure* indicate non-zero formal charges next to each atom i.e. (+1)	**Electron Pair Geometry around each central atom**	**Molecular Geometry around each central atom**	**Bond Angles (BA) and Bond Orders (BO)**	**Polar or Non-Polar Molecule?**	**Hybridization around each central atom**	**Build the Molecule and get Instructor's Initials below**
HCN							
SF_6							
C_2H_4							

	Best Lewis Structure* indicate non-zero formal charges next to each atom i.e. (+1)	**Electron Pair Geometry around each central atom**	**Molecular Geometry around each central atom**	**Bond Angles (BA) and Bond Orders (BO)**	**Polar or Non-Polar Molecule?**	**Hybridization around each central atom**	**Build the Molecule and get Instructor's Initials below**
XeF_4							
I_3^-							
CH_3Br							

	Best Lewis Structure* indicate non-zero formal charges next to each atom i.e. (+1)	**Electron Pair Geometry around each central atom**	**Molecular Geometry around each central atom**	**Bond Angles (BA) and Bond Orders (BO)**	**Polar or Non-Polar Molecule?**	**Hybridization around each central atom**	**Build the Molecule and get Instructor's Initials below**
C_2H_2	Both carbons are central.						
C_6H_6	The six C atoms form a ring.						
BrF_5							

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Sample Laboratory Report

Compound Stoichiometry: Determining the Formula of an Oxide of Aluminum and the Hydration Number of a Transition Metal Hydrate

September 15, 2015

Student: Annie Wan Qanduit
Partner: Will Helper

CHEM 111-03
Tuesday 3:00 – 5:50 PM
Professor Bishop

Purpose: The objective of Part A was to determine the compound formula of an aluminum oxide. The objective of Part B was to determine the hydration number of a hydrated transition metal compound.

Procedure: The procedure given in the SUNY Oneonta General Chemistry I Laboratory Manual, Revision 8-15, pp 28 – 32, was used. No modifications were made.

Observations and Data:

Part A: The aluminum powder used for this part had a dull gray appearance. When it was heated in the crucible, crackling was heard over the sound of the burner flame. After heating, the material in the crucible was a white powder.

Data for the three trials are summarized in the following table:

Table 1: Data from Part A

Mass of:	Trial 1	Trial 2	Trial 3
Crucible + lid	47.524 g	48.312 g	48.107 g
Crucible + lid + Al	47.925 g	48.717 g	48.520 g
Aluminum alone	0.401 g	0.405 g	0.413 g
After 1st heating	48.236 g	48.953 g	48.776 g
After 2nd heating	48.276 g	49.075 g	48.891 g
After 3rd heating	48.279 g	49.077 g	48.889 g
Oxide alone*	0.755 g	0.765 g	0.782 g

*Difference of total mass before and after heating.

Part B: The hydrated cobalt(II) chloride used for this part appeared as coarse crystals which had a red-purple color. After heating, the crucible contained a fine light-blue powder with a few dark-blue flecks.

Data for the three trials are summarized in the following table:

Table 2: Data from Part B

Mass of:	Trial 1	Trial 2	Trial 3
Crucible + lid	47.524 g	48.312 g	48.107 g
Crucible + lid + hydrate	49.522 g	50.325 g	50.110 g
Hydrated salt alone	1.998 g	2.013 g	2.003 g
After 1st heating	48.756 g	49.788 g	49.662 g
After 2nd heating	48.695g	49.492 g	49.279 g
After 3rd heating	48.689 g	49.487 g	49.274g
Water driven off*	0.833 g	0.838 g	0.836 g

*Difference of total mass before and after heating.

Calculations and Results:

Part A, Trial 1:

0.755 g oxide – 0.401 g Al = 0.354 g O.

0.401 g Al x 1mol/26.9815 g = 0.0149 mol Al

0.354 g O x 1 mol/15.9994 g = 0.0221 mol O

Ratio oxygen/aluminum = 0.02213 mol/0.01486 mol = 1.49

Calculations for all three trials are summarized in the following table:

Table 3: Calculated Results for Part A

Value	Trial 1	Trial 2	Trial 3
mass Al	0.401 g	0.405 g	0.413 g
mass O	0.354 g	0.360 g	0.369 g
mol Al	0.0149 mol	0.0150 mol	0.0153 mol
mol O	0.0221 mol	0.0225 mol	0.0231 mol
mole ratio O/Al	1.49	1.50	1.51

The average O/Al mole ratio is (1.49 + 1.50 + 1.51)/3 = 1.50. At exactly halfway between 1 and 2, a multiplier is needed to produce an integer ratio. Multiplying by a factor of 2 produces the ratio 3 O/2 Al.

The formula for aluminum oxide appears to be Al_2O_3.

Part B, Trial 1:

1.998 g hydrate – 0.833 g water = 1.165 g anhydrous $CoCl_2$.

1.165 g $CoCl_2$ x 1 mol/129.84 g = 0.008973 mol $CoCl_2$

0.833 g H_2O x 1 mol/18.015 g = 0.0462 mol H_2O

Ratio water/cobalt(II) chloride = 0.04624 mol/0.008973 mol = 5.15

Calculations for all three trials are summarized in the following table:

Table 4: Calculated Results for Part B

Value	Trial 1	Trial 2	Trial 3
mass $CoCl_2$	1.165 g	1.175 g	1.167 g
mass H_2O	0.833 g	0.838 g	0.836 g
mol $CoCl_2$	0.00897 mol	0.00905 mol	0.00899 mol
mol H_2O	0.0462 mol	0.0465 mol	0.0464 mol
mol ratio $H_2O/CoCl_2$	5.15	5.14	5.16

The average $H_2O/CoCl_2$ mole ratio is (5.15 + 5.14 + 5.16)/3 = 5.15. This is much nearer to 5 than to 6.

The formula for cobalt(II) chloride hydrate appears to be $CoCl_2 \cdot 5\ H_2O$.

Discussion: Our experimental evidence from Part A indicates that aluminum oxide has the formula Al_2O_3. According to Wikipedia (http://en.wikipedia.org/wiki/Aluminium_oxide), this is its actual formula. Our experimental evidence from Part B indicates that cobalt chloride hydrate has the formula $CoCl_2 \cdot 5H_2O$. According to Wikipedia (http://en.wikipedia.org/wiki/Cobalt(II)_chloride), cobalt chloride has two hydrated forms, a dihydrate and a hexahydrate. The hexahydrate is reddish-purple (like the starting material for this lab) and the dihydrate is deep blue, like the flecks in our heated samples. Therefore, our results for Part B were off by one hydration unit.

Possible sources of error could have included allowing some material to escape the crucibles as they were heated, weighing the crucibles before they were completely cooled to room temperature, and failing to heat them long or hard enough. For Part A, these error sources appear to have been well controlled. However, in Part B, it appears that the samples were not heated enough to drive off all the water of hydration, as flecks of dihydrate were observed (but not recognized at the time). Consulting the online literature about reactants and products before actually doing the lab may have helped us to obtain better results.

Inside of Back Cover

Periodic Table of the Elements