

SUNY COLLEGE AT ONEONTA

General Chemistry I

Lab Manual

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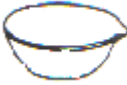






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Department of Chemistry and Biochemistry

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).

Overview of Laboratory Glassware		
This is a guide illustrating the different types of glassware and chemistry utensils you will use this semester and how to use them properly.		
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.
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 or make solutions in. It has slanted walls so it's easier to swirl your reaction.
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.
Crucible Tong		Use this to transfer a hot crucible and cover off of a pipestem triangle. Do not use this to transfer other glassware, it is not designed to grip other glassware.
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.

<p>Ring Stand</p>		<p>Set up various clamps to this to hold your glassware in place.</p>
<p>Ring Clamp</p>		<p>Clamp this to the ring stand and place a wire gauze on top to have a level surface to place other glassware for heating.</p>
<p>Utility Clamp</p>		<p>Use this to clamp on to various glassware such as test tubes or Erlenmeyer flasks.</p>
<p>Wire Gauze</p>		<p>Use this in combination with the ring clamp to provide a level surface for heating glassware.</p>
<p>Bunsen Burner</p>		<p>Use this to heat solutions. There are generally two controls on a Bunsen burner; one will control the amount of gas being let in and the other will control the amount of air let in.</p>
<p>Crucible and Cover</p>		<p>Use this to heat a compound intensely – usually with the intent of melting or dehydrating it.</p>
<p>Pipestem Triangle</p>		<p>Use this as a support in combination with the ring clamp for a crucible and cover.</p>

How to Prepare a Lab Report

This document explains how you should prepare lab reports for most of the laboratory experiments in this course. This format should be used unless you are told otherwise.

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; just cite it from the lab handout and note any changes. If there are changes to the lab during the course of the lab session, write it on your Data Sheet so you don't forget.

Observations and Data – This section should include all the raw data that you took during lab. Anything that is important for completing calculations should be shown in the “raw data” format in this section. It may be helpful to put your data in table formats. When recording data do not round, enter all the decimal places indicated unless otherwise specified. If you made observations of chemical/physical changes during the lab, you should not them here as well.

Calculations – You should show all of your work for any calculations you make for a lab. 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.

Conclusions – Reflect back on your Purpose of the lab. Explain what you can conclude about the Purpose based on the Observations, Data and Calculations. This should be at least a few sentences in length.

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 below.

Lab Report Format:

Name of Experiment

Name _____

Date

Lab Section _____

Purpose:

(~2 sentences)

Procedure:

(Cite the lab manual and note any changes to procedure)

Observations and Data:

(Include all raw data, observations; put information in table format if you can)

Calculations:

(Show all work)

Conclusions:

(At least a couple sentences reflecting on the purpose)

Questions:

(Rewrite the questions, answer and number them)

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^-			
INSOLUBLE COMPOUNDS		EXCEPTIONS	
Most salts of carbonate, CO_3^{2-} phosphate, PO_4^{3-} oxalate, $\text{C}_2\text{O}_4^{2-}$ 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-}		$\text{Ba}(\text{OH})_2$ is soluble	

Solubility Rules

all acids are soluble

Strong Acids	Strong Bases
HCl	LiOH
HBr	NaOH
HI	KOH
HNO_3	$\text{Ca}(\text{OH})_2(\text{s})$
HClO_4	$\text{Ba}(\text{OH})_2(\text{s})$
H_2SO_4	

Weak Acids	Weak Bases
CH_3COOH	CH_3COO^-
NH_4^+	NH_3
H_2CO_3	CO_3^{2-}
$\text{H}_2\text{C}_2\text{O}_4$	$\text{C}_2\text{O}_4^{2-}$
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:

$$2\text{H}^+ + \text{CO}_3^{2-} \longrightarrow \text{H}_2\text{O}(\text{l}) + \text{CO}_2(\text{g})$$

$$2\text{H}^+ + \text{S}^{2-} \longrightarrow \text{H}_2\text{S}(\text{g})$$

$$2\text{H}^+ + \text{SO}_3^{2-} \longrightarrow \text{H}_2\text{O}(\text{l}) + \text{SO}_2(\text{g})$$

$$2\text{H}^+ + \text{M} \longrightarrow \text{M}^{2+} + \text{H}_2(\text{g})$$

M = a metal atom

Strong Electrolytes:
Soluble ionic compounds
Strong acids and strong bases

Determining Net Ionic Equations

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

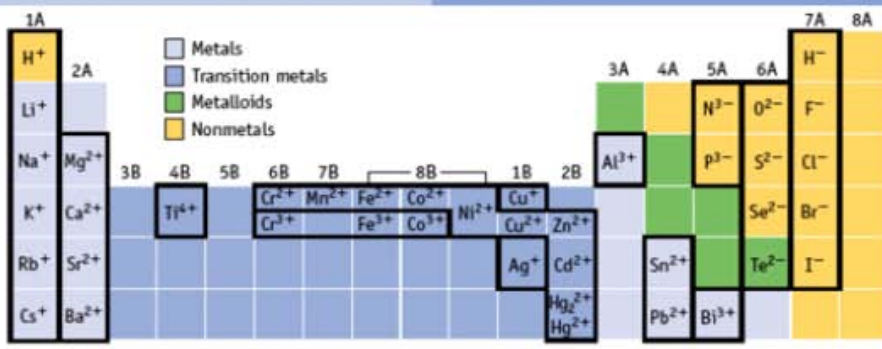


Table 3.1 Formulas and Names of Some Common Polyatomic Ions

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	$\text{Cr}_2\text{O}_7^{2-}$	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)		

Viewing Moles Lab

Objective:

This short laboratory exercise is intended to give you a good feeling for the size of a mole.

Hazards:

Ethanol is extremely flammable. Wear goggles at all times!

Introduction:

The mole is the central chemical counting unit. Atoms and molecules are small, so you need lots of them present in order to see or even measure them accurately. The mole is a number that brings atoms and molecules to a usable human scale. The mole is based on Avogadro's number.

$$1 \text{ mole} = 6.022 \times 10^{23} \text{ things}$$

To have a mole of something, it does not matter what it is but just how many of them are present. A mole of apples is 6.022×10^{23} apples and a mole of grapes is 6.022×10^{23} grapes. It is clear that if you have the same number of apples and grapes, the volume of the apples will be the larger of the two, and the mole of apples will weigh more than the mole of grapes.

Molar Mass is measured in grams/mole. The Molar Mass of a compound tells you how many grams you would have to weigh out to obtain one mole of the substance.

Procedure:

Work in pairs for this exercise. Each one of you should measure out one mole of the following, using either a graduated cylinder or the balance:

Compound/Element (Formula)	Molar Mass	Density	Observations
Water (H ₂ O)	18 g/mol	1.00 g/ml	
Ethanol (C ₂ H ₆ O)	46 g/mol	0.78 g/ml	
Sodium chloride (table salt) (NaCl)	58 g/mol	-	
Sodium carbonate (Na ₂ CO ₃)	106 g/mol	-	
Sand (SiO ₂)	60 g/mol	-	
Copper (Cu)	64 g/mol	-	
Sugar (sucrose) (C ₁₂ H ₂₂ O ₁₁)	342 g/mol	-	

Chemical Synthesis and Growing Crystals

Objective:

To synthesize (that means to prepare from other reagents), a chemical compound with the formula $K_3[Fe(C_2O_4)_3] \cdot 3H_2O$ (Figure 1).

Hazards:

Sulfuric acid and oxalic acid are corrosive. Wear goggles at all times!

Introduction:

The molecule you will be synthesizing contains an Fe^{3+} ion in the center with three $C_2O_4^{2-}$ ions attached to it like blades of a propeller. This unit, called a *complex ion*, has an electrical charge of 3-, so there are three potassium ions (K^+) in the solid to balance this negative charge. In addition, when the compound forms solid, green crystals, three water molecules are trapped in the solid for each unit of $K_3[Fe(C_2O_4)_3] \cdot 3H_2O$.

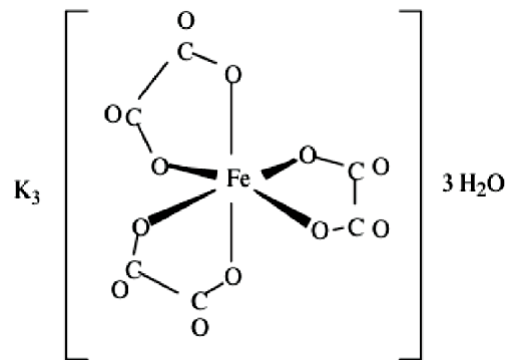


Figure 1: $K_3[Fe(C_2O_4)_3] \cdot 3H_2O$
This is a beautiful green, crystalline solid.

Procedure:

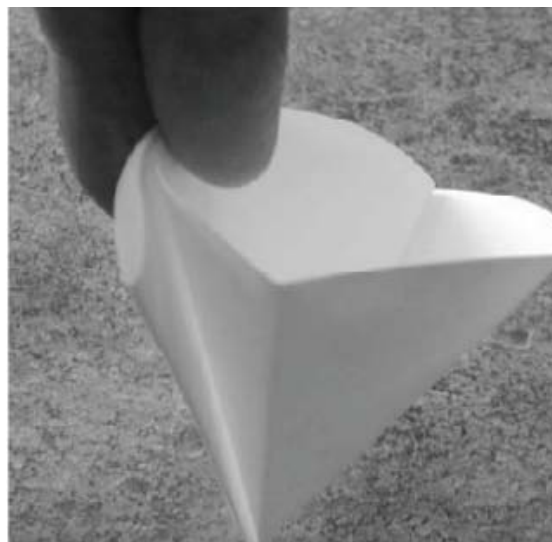
1. Add 5 drops of dilute sulfuric acid (H_2SO_4) to 15ml of warm water in a 250ml beaker. Next add 5g of ferrous ammonium sulfate hexahydrate, $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$. When everything has been dissolved add 25ml of 1.0M oxalic acid ($H_2C_2O_4$), and then heat the solution to boiling; stir constantly.
2. Allow the yellow precipitate (the solid) to settle, and then *decant* (carefully pour off) the liquid. Wash the precipitate with distilled water, and then decant the liquid after the precipitate has settled again. Throw the liquid away down the drain with lots of water, keep the solid.
3. Add 10ml of saturated, aqueous potassium oxalate ($K_2C_2O_4$) to the washed solid precipitate. Heat the solution to $40^\circ C$, and then add 20ml of 3% hydrogen peroxide (H_2O_2), a few milliliters at a time, stirring continuously. Wait a few moments between additions of hydrogen peroxide to keep the temperature near $40^\circ C$.
4. Ignoring any red-brown precipitate [$Fe(OH)_3$] that may form, heat the solution to boiling, and add 5ml of 1.0M oxalic acid. Add another 3ml of the oxalic acid solution dropwise (one drop at a time), keeping the solution near boiling.
5. Gravity filter the hot solution into a 100ml beaker as demonstrated by your instructor and on the following page.
6. Add 10ml of ethanol (ethyl alcohol) and warm to dissolve any crystals that may form.
7. Tie a short string to a splint and suspend the thread in the solution. Place a piece of paper over the beaker labeled with your name(s) and store in your drawer (or designated area) until next lab.
8. In the next laboratory, remove the thread, now covered with crystals, from the beaker. Transfer the crystals to a paper towel and blot dry. If crystals line your beaker, decant the liquid off of them, and then use a spatula to scrape the crystals out onto a paper towel. Once blotted dry, weigh them and record the weight on the report form. Dispose of the crystals as explained by your instructor.

Preparing a Gravity Filtration

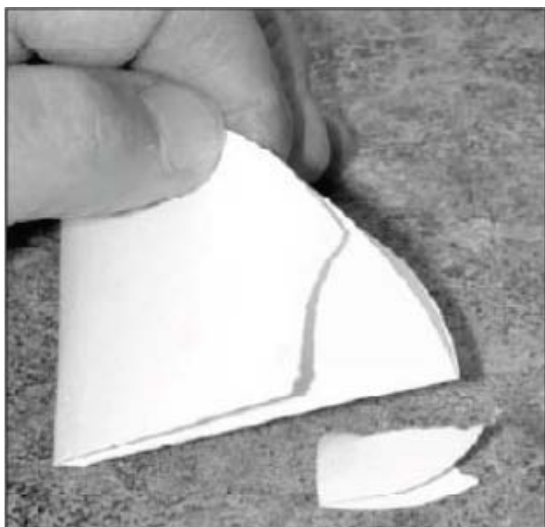
Filtration is used to separate a solid from a mixture of that solid suspended in a liquid or a solution. The simplest filtration method used is to pour the mixture through a filter. The solids get stuck in the filter and the liquid phase passes through. In order to obtain as pure a solid as possible,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



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



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



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

Viewing Moles and Growing Crystals Data Sheet

On this sheet, write all observations, data, and procedural changes that occur in the lab. Observations should include any descriptions of materials and chemical/physical changes. Data should include all weighed masses, any recorded temperature, times, pH values, calculations, etc.

Analysis of a Mixture

Objective:

To determine the percent composition of a mixture containing unknown proportions of

Substance A : Barium chloride dehydrate ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$)

Substance B: Anhydrous barium chloride (BaCl_2)

Note that substances A and B are *completely* different. Do NOT mix up your understanding of which is which.

Hazards:

The crucible and cover will be extremely hot once heated, use crucible tongs to handle properly. Wear goggles at all times.

Introduction:

In this reaction, the only difference between substances A and B are that one of them (A) has two water molecules attached. You will be heating the mixture to dehydrate substance A, barium chloride dehydrate. This will lead to the loss of the waters of hydration from the $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$. The anhydrous BaCl_2 (substance B) is not affected by this procedure.

The only thing that changes in this procedure is the removal of the two water molecules from substance A, $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$.

Your goal is to identify the percent of each compound in the mixture. The idea here is that by performing the procedure given and taking appropriate measurements at the appropriate points, you can determine everything you need to arrive at the percentages of substance A and B.

Procedure:

Before you begin you must decide what you will measure during the experiment and how that will lead to the percentages you are looking for. Read through the procedure then write what you will measure on the Data Sheet for this lab and show it to your instructor before you begin.

1. Preparation of the Crucible – Thoroughly clean and dry your porcelain crucible and its cover. To ensure dryness, place the crucible and its cover on the triangle and heat gently for 5 minutes, brushing the flame back and forth over the bottom of the crucible. Let the apparatus cool completely to room temperature before using.
2. Obtain an unknown sample and weigh out about 2 grams. Record your actual mass on your Data Sheet and your Unknown Number.



Figure The bottom of the porcelain crucible will glow cherry red when it is heated properly.

3. Place your crucible with the sample in the triangle mounted on the ring stand.
4. Adjust the height of the ring so that the bottom of the crucible is only a few centimeters from the top of the barrel. Displace the cover carefully slightly to one side to allow water vapor to escape.
5. Light the burner and, holding the burner at its base, heat gently by brushing the flame across the bottom of the crucible for about 10 minutes.
6. After the initial heating, place the burner directly under the crucible and heat the bottom of the crucible until it glows cherry red (some may not glow as intensely as others). Heat in this manner for at least 5-6 minutes, then remove the flame and allow the assembly to cool to room temperature.
7. When the crucible is no longer warm to the touch, the dehydration is complete.
8. Repeat steps 2-7 with a second sample of unknown.
9. Calculate the percentage of mass lost during the reaction for the two experiments. If they differ by more than 10%, then repeat the experiment a third time.

Report:

Complete 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.

Analysis of a Mixture Data Sheet

On this sheet, write all observations, data, and procedural changes that occur in the lab. Observations should include any descriptions of materials and chemical/physical changes. Data should include all weighed masses, any recorded temperature, times, pH values, calculations, etc.

Synthesis of Alum: $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$

Objective:

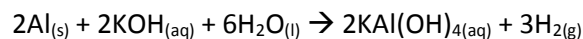
To use aluminum foil to synthesize a chemical compound, alum, which is hydrated potassium aluminum sulfate, $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$.

Hazards:

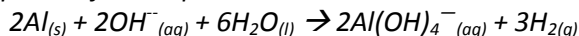
- Potassium hydroxide is a strong, corrosive base. If you get in on you your skin will feel slippery, rinse with lots of water.
- Sulfuric acid is a strong, corrosive acid. If you get it on you your skin will burn, rinse with lots of water.
- You will be generating H_2 gas (hydrogen gas), it is VERY flammable, do this step in the HOOD!
- Wear goggles 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" $[\text{Al}(\text{OH})_4^-]$. This can be described by the following complete, balanced equation:



The Net Ionic Equation for this process is:

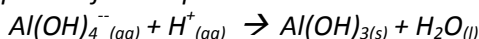


After filtration to remove any unwanted material, the alkaline solution of $\text{Al}(\text{OH})_4^-$ is **clear** and **colorless**. H_2 gas is evolved, which mixes with the atmosphere. The chemical species that remain in solution are potassium ions (K^+) and aluminate ions $[\text{Al}(\text{OH})_4^-]$,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

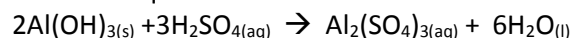
In the next step, sulfuric acid is added and two sequential reactions occur. Initially, before the addition of acid is complete, insoluble aluminum hydroxide is formed $[\text{Al}(\text{OH})_3]_{(s)}$. It appears as a **thick, white, gelatinous precipitate**. This reaction is described by:



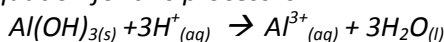
The Net Ionic Equation for this process is:



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



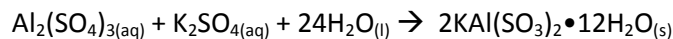
The Net Ionic Equation for this process is:



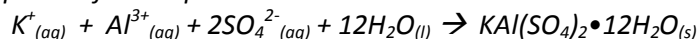
At this point, the solution contains Al^{3+} ions, K^+ ions, and SO_4^{2-} ions. On cooling, crystals of hydrated potassium aluminum sulfate, $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ (or alum) are very slowly deposited. In the experiment,

the crystallization process is sped up 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.

The full equation for the formation of alum is below:



The Net Ionic Equation for this process is:



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.

Procedure:

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.

Before starting, read through the procedure and determine where you have to record accurate weights. Write this on your Data Sheet and show your instructor before beginning.

1. Make sure all glassware is clean and dry before beginning this experiment.
2. Obtain about 1g of aluminum foil (about a 1-inch strip of heavy duty aluminum foil)
3. Weigh the exact mass of foil, then cut it into small pieces and place it in a 250mL beaker.
4. Be careful not to lose any pieces of metal.
5. DO THIS STEP IN THE HOOD! Using a graduated cylinder, add 50ml of 1.4M KOH.
6. Set up a ring stand, ring clamp, wire gauze, and Bunsen burner. Place the beaker on the gauze and heat the beaker over a low flame. It is not necessary to boil the solution. The aluminum will take about 10-15 minutes to dissolve.
7. While the aluminum is dissolving, set up an apparatus for **gravity filtration** (see the previous Growing Crystals experiment for this set up). Place a clean funnel with the piece of folded filter paper on a 125ml Erlenmeyer flask.
8. When the aluminum has dissolved (as evidence by the lack of H₂ bubbles given off), 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.
9. Allow the flask to cool. While cooling, wash the funnel and beaker with lots of tap water to remove any KOH left in there.
10. When the solution is reasonably cool, add 20ml of 9M H₂SO₄ (measure with a graduated cylinder in the hood), quickly and with care. 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 H₂SO₄, place the flask back over the Bunsen burner and warm it gently while swirling until all of the solid has dissolved.
11. Make an ice bath by putting ice and a little water in a large beaker (~600ml)
12. Allow the flask to cool until just warm, then put it in the ice bath for at least 5 minutes.
13. 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 minutes undisturbed.
14. While the solution is cooling, pour 50mL of the 50% alcohol/water mixture into a test tube or clean graduated cylinder and cool it in the ice bath.

15. Set up a **vacuum filtration** apparatus (Buchner funnel, see figure below) as demonstrated by your instructor. Pour a small amount of water on the filter paper.
16. 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.
17. Pour about 10ml 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.
18. When done filtering, place your crystals and filter paper in a beaker or on a watch glass, label them and store them in your drawer or other designated area until next week to ensure they are completely dry.
19. Scrub all glassware and the Buchner funnel, and then return any borrowed supplies to the appropriate part of the lab.

Next Week:





Weigh your alum crystals, and then complete the calculations for theoretical yield and percent yield using the actual mass of reagent and products you weighed out in lab. Waste goes in the designated container

Report:

Complete 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.

Preparing a Vacuum Filtration

 <p>Filter flask</p> <p>Filter funnel</p> <p>Filter paper</p>	<p>Step 1: You need a glass or plastic filter flask (with sidearm), a plastic or porcelain funnel, and a piece of filter paper.</p>	 <p>Clamp flask onto ringstand</p>	<p>Step 2: Clamp the filter flask onto your ringstand so the flask will not tip over. Attach a piece of rubber vacuum tubing.</p>
 <p>Plastic fitting on vacuum tubing</p>	<p>Step 3: At the other end of the vacuum tubing make sure there is a little plastic fitting.</p>		<p>Step 4: Plug the vacuum tubing with the plastic fitting onto the water spigot in front of your lab station. Turn on the water all the way and test to see if a vacuum has been created in your flask and funnel.</p>

Synthesis of Alum Data Sheet

On this sheet, write all observations, data, and procedural changes that occur in the lab. Observations should include any descriptions of materials and chemical/physical changes. Data should include all weighed masses, any recorded temperature, times, pH values, calculations, etc.

Analysis of Silver Group Cations (Ag^+ , Pb^{2+} , Hg_2^{2+})

Objective:

To observe reactions between silver group ions (Ag^+ , Pb^{2+} , and Hg_2^{2+}) and reagents; then use those observations to separate a mixture of silver group ions based on their properties.

Hazards:

- When working with the centrifuges, they must be BALANCED! That means approximately the same amount of liquid and the same type of test tube across from each other.
- Do NOT put waste down the drains! All heavy metal waste must be put in the designated container in the HOOD!

Introduction:

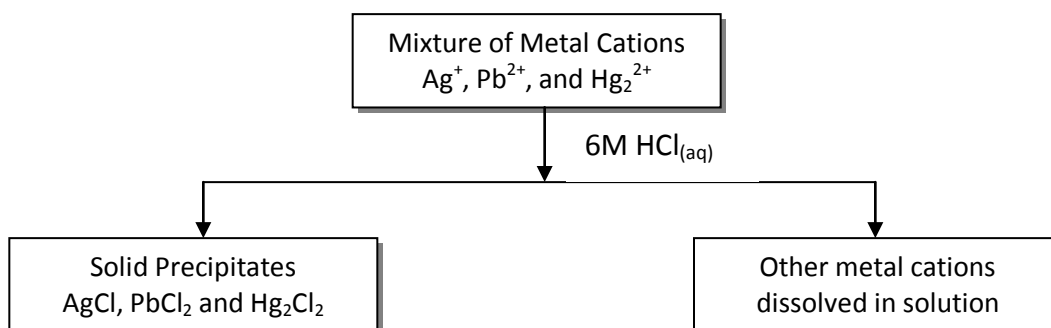
One problem often faced in qualitative analysis is to test for one ion in a mixture of many ions. To find a test for one ion that is not interfered with by another ion is nearly impossible. Therefore, if one has a mixture of a large number of ions, the usual approach is to use a chemical method to separate the mixture into subgroups that consist of just a few ions. Then it may be possible to test for one particular ion in the presence of just one or two others. Alternatively, each subgroup of just a few ions may be separated further so that each ion in the subgroup ends up in a different test tube where its presence can be confirmed by other chemical tests.

Precipitation reactions are of particular importance in qualitative analysis, and they are important in the silver group. In addition, acid-base, complexation, and even oxidation-reduction reactions are useful.

In this experiment, you will focus on how to separate the **silver group ions** – silver(I) Ag^+ , lead(II) Pb^{2+} , and mercury(I) Hg_2^{2+} – using basic reactions, especially precipitation, acid-base, and complex formation. Then you will confirm the presence of each in solution.

CHEMISTRY OF THE SILVER GROUP CATIONS

These three cations – silver(I) Ag^+ , lead(II) Pb^{2+} , and mercury(I) Hg_2^{2+} – are used in this experiment because they are the only common metals to form insoluble precipitates with chloride ions. This means that when mixed with 6M HCl, each one of these ions would make its respective chloride salt.



Once the three silver cations are separated from all other ions, they can be separated by chemical means based on their different chemical properties.

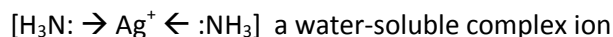
Compound	Color	Solubility per 100g H ₂ O at 20°C	Solubility per 100g H ₂ O at 100°C	Reaction with Ammonia
AgCl	White	0.00015g	0.0021g	Forms colorless soluble complex
PbCl ₂	White	1.0g	3.3g	Forms white ppt. Pb(OH)Cl
Hg ₂ Cl ₂	White	0.0002g	0.001g	RedOx Reaction

Imagine you have a mixture of all three solid chlorides and you need to separate them.

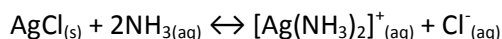
From the table, we see that **PbCl₂** is the most soluble in hot water, so the first step in separating the mixture of ions will be to treat the mixture with hot water to selectively dissolve the PbCl₂. The AgCl₂ and Hg₂Cl₂ will remain as solids. Separate the liquid with Pb²⁺ in it from the solids, and treat the liquid with potassium chromate, K₂CrO₄. This will produce a bright yellow solid if lead is present:



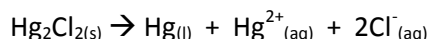
Now we have to deal with the mixture of **AgCl₂** and **Hg₂Cl₂**. We take advantage of the tendency of transition metals, such as Ag⁺, to form water-soluble complex ions with ammonia. Here, the ammonia uses the lone pair of electrons of the N atom to form a bond with the Ag⁺ ion:



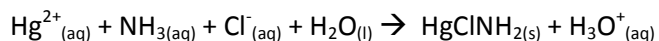
When a large enough concentration of NH₃ is added to an insoluble precipitate of **AgCl**, the ammonia binds to the Ag⁺ ion and forms the complex ion. The net result is that the AgCl dissolves:



Now the silver ion is in the liquid, and the **Hg₂Cl₂** is still a solid. The relatively concentrated ammonia is added to Hg₂Cl₂ it can undergo what is known as a disproportionation reaction. That is, the mercury(I) ion is both oxidized to mercury(II) and reduced to metallic mercury.



This reaction is induced by the presence of ammonia. In addition, when ammonia and Cl⁻ are present, the Hg²⁺ ion forms a rather strange compound, an amido salt HgClNH₂. The latter is an insoluble white solid.

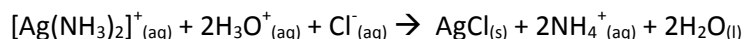


Therefore, when NH₃ is added to the AgCl/Hg₂Cl₂ mixture, the AgCl dissolves, and the Hg₂Cl₂ turns into black or gray finely divided mercury metal and white insoluble HgClNH₂. The net reaction for Hg₂Cl₂ is:



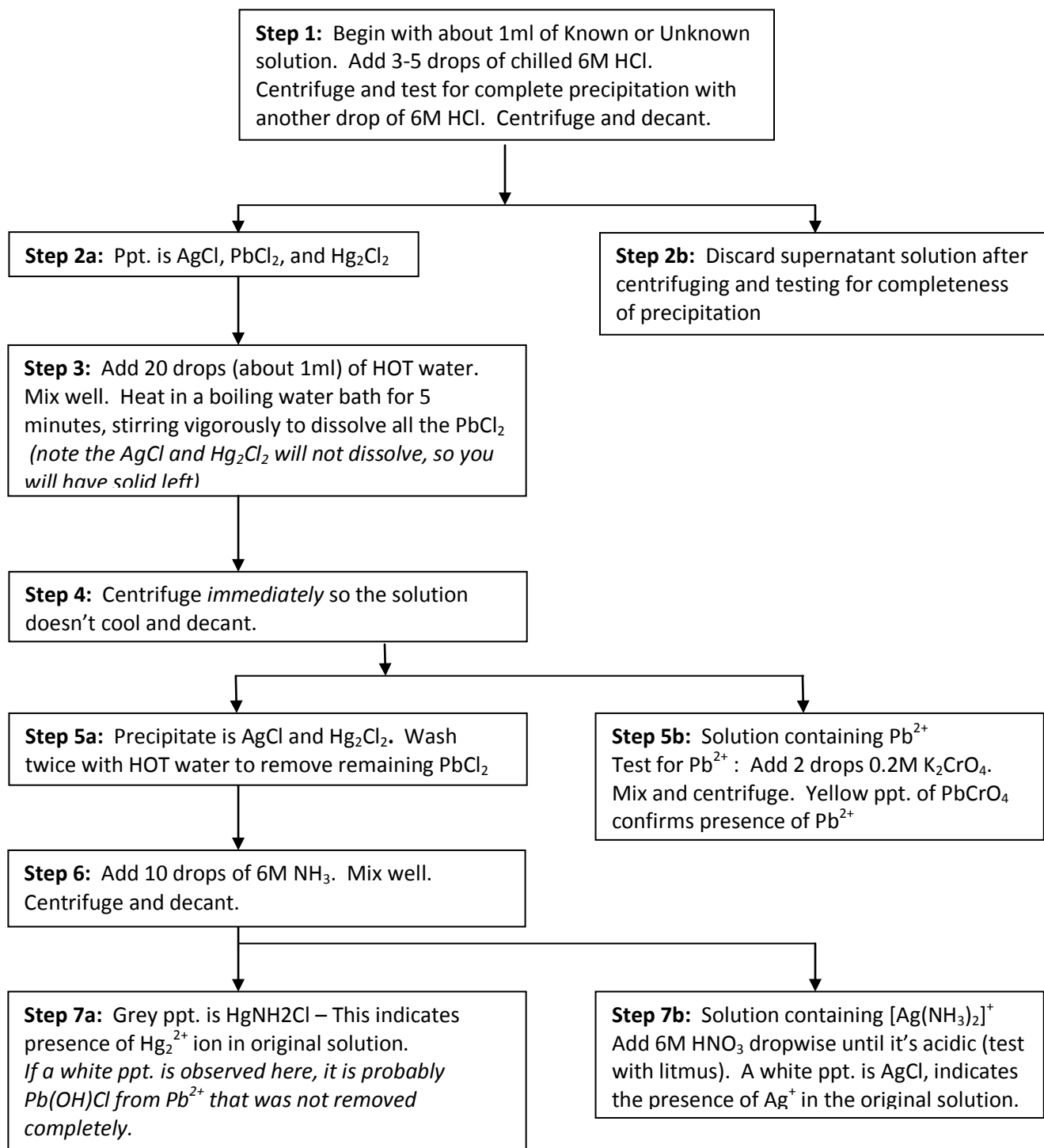
Notice that a second molecule of the base NH₃ is used to “collect” the H⁺, an acid, that is produced with Hg²⁺ reacts with NH₃, the product is of course the salt NH₄Cl. The importance of this reaction is that it provides confirmation of the presence of the mercury(I) ion, Hg₂²⁺, in a solution of unknown composition.

The only step left is to confirm the presence of Ag⁺. Take the solution containing the complex silver ion and add nitric acid. The nitric acid reacts with the NH₃ (a base) to form a very stable ammonium ion. NH₃ is no longer bound to Ag⁺, and since Cl⁻ is still present, it will react with the Ag⁺ to reform the white solid precipitate, AgCl, formation of this white solid is confirmation of the presence of Ag⁺.



Procedure:

To understand the chemistry of each ion, you will first analyze a sample with ALL THREE silver group ions in it, this is the Known solution. Then you will receive an Unknown solution with 1, 2, or 3 silver group ions in it. You will be able to determine the ions present in your unknown based on your observations. You will use the flow chart below as a procedure. Each time you change your solution in some way, you will write your observations on the Data Sheet page. Keep your observations for the Known and Unknown solutions separate.



Report:

Complete 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.

You must include your Unknown Number and the identification of the ions present and absent from your unknown. Also, type your observations from analyzing your Unknown.

Answer the following *after* the Conclusion:

1. Write the balanced, Net Ionic Equation for the reaction occurring when
 - a. Cl^- is added to a solution containing Pb^{2+}
 - b. Cl^- is added to a solution containing Hg_2^{2+}
2. Write a balanced equation for the reaction that occurs on adding excess aqueous ammonia to $\text{AgCl}_{(s)}$.

Analysis of Silver Group Cations Data Sheet

On this sheet, write all observations, data, and procedural changes that occur in the lab. Observations should include any descriptions of materials and chemical/physical changes. Data should include all weighed masses, any recorded temperature, times, pH values, calculations, etc.

Known Solution Observations (contains all 3 ions):

Unknown Solution Observations (Unknown Number _____):

Determination of the Molar Mass of an Unknown Diprotic Acid

Objective:

To standardize (determine precisely the concentration of) a solution of the base sodium hydroxide, NaOH, using oxalic acid dehydrate, $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$, as a primary standard acid. Then use the sodium hydroxide, with its now known concentration, to titrate an unknown acid, and then use the titration information to solve for the acid's molar mass.

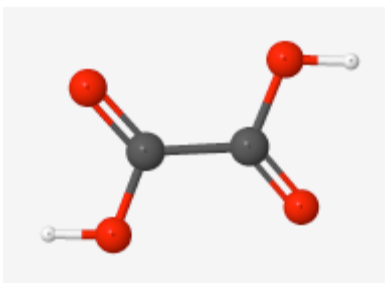
Hazards:

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

Introduction:

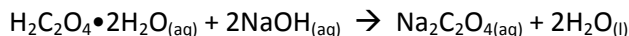
Titration is the process for ascertaining the exact volume of one solution that is chemically equivalent to a given amount of another substance, either another solution or a given amount of solid material dissolved in a solvent. The apparatus usually used in titrations is a burette. If a solution of an acid is titrated with a solution of a base, the **equivalence point**, the point at which chemically equivalent quantities of acid and base have been mixed, can be found by means of an indicator.

In this experiment, you must first standardize the base, NaOH, with a primary standard acid, $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$. 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.



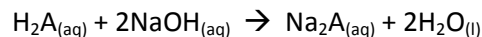
Oxalic acid, $\text{H}_2\text{C}_2\text{O}_4$, has two ionizable H atoms, so two moles of NaOH are required to consume one mole of acid.

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



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 dehydrate 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:



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:

$$\text{Moles of NaOH} = \text{Concentration of NaOH (mol/L)} \times \text{Volume of NaOH (L)}$$

The moles of NaOH consumed are related to the moles of the unknown diprotic acid through stoichiometric factor (of $\frac{1}{2}$, 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

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 250ml of distilled water into a 500ml Erlenmeyer flask. Add 50ml of 3M NaOH (NOTE – the total amount of solution is now 300ml!) Stopper the flask and shake well.
3. Rinse your burette once more with distilled water and then a few times with small (5-10ml) 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.
4. In the *Prelab Assignment* you should have calculated the mass of $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ required for the standardization of 25mL of your diluted NaOH solution. On a piece of weighing paper, weigh out the appropriate amount of oxalic acid and record all decimal places.
5. Place the sample in a clean 250mL flask, dissolve it in about 50mL of distilled water, add two drops of phenolphthalein indicator and record the initial burette reading to at least two decimal places (estimate the last one). Place your Erlenmeyer flask with the acid in it under a white piece of paper. 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.

6. Titrate at least one more sample of the standard acid, 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!

Determination of the Molar Mass of an Unknown, Diprotic Acid

1. Obtain two samples of the same unknown acid; each should be about 1gram. Label each weighing paper with the mass of the acid that is on it so you don't mix up your samples. Record the unknown number and the mass of each trial on your Data Sheet.
2. Add 50ml of distilled water and 2 drops of phenolphthalein to each Erlenmeyer flask, and then titrate with the standardized NaOH solution to a permanent light pink endpoint. Your unknown sample may not completely dissolve with you add distilled water, but the solution process should be completed as you add NaOH; make sure the entire solid is dissolved by the endpoint.

Report:

Complete 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.

You must include your unknown number in your lab 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.

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.

Determination of the Molar Mass of an Unknown Diprotic Acid Data Sheet

On this sheet, write all observations, data, and procedural changes that occur in the lab. Observations should include any descriptions of materials and chemical/physical changes. Data should include all weighed masses, any recorded temperature, times, pH values, calculations, etc.

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.

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 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.

What to record:

For each mixture, you should record what you see happen. 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 in the Net Ionic equations. All observations will be written in the boxes provided; there will not be a Data Sheet for this lab.

What to do next:

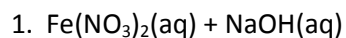
For each mixture that does react in some way, write the overall equation and the Net Ionic equation. Both need to be balanced and you need to write the states for each compound, ion, and/or element in the reaction. **Work individually to perform the reactions, but team up to figure out the reaction equations.** It is up to your instructor if you will hand the packet in at the end of lab or the following lab session.

Report:

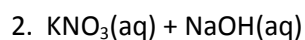
You will NOT have a separate write-up for this lab. You will be handing in the packet with all of the observations and equations on it as a lab report.

Net Ionic Equations Lab

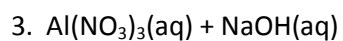
Name _____



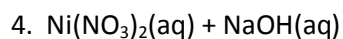
Observations	
Overall Equation	
Net Ionic Equation	



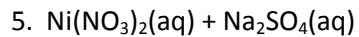
Observations	
Overall Equation	
Net Ionic Equation	



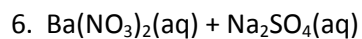
Observations	
Overall Equation	
Net Ionic Equation	



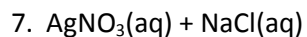
Observations	
Overall Equation	
Net Ionic Equation	



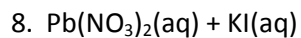
Observations	
Overall Equation	
Net Ionic Equation	



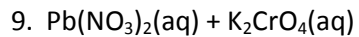
Observations	
Overall Equation	
Net Ionic Equation	



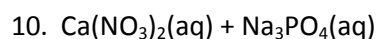
Observations	
Overall Equation	
Net Ionic Equation	



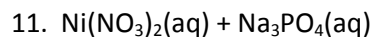
Observations	
Overall Equation	
Net Ionic Equation	



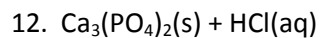
Observations	
Overall Equation	
Net Ionic Equation	



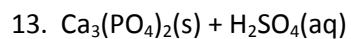
Observations	
Overall Equation	
Net Ionic Equation	



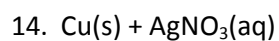
Observations	
Overall Equation	
Net Ionic Equation	



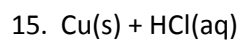
Observations	
Overall Equation	
Net Ionic Equation	



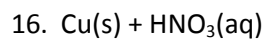
Observations	
Overall Equation	
Net Ionic Equation	



Observations	
Overall Equation	
Net Ionic Equation	



Observations	
Overall Equation	
Net Ionic Equation	



Observations	
Overall Equation	
Net Ionic Equation	

17. $\text{Zn(s)} + \text{HCl(aq)}$

Observations	
Overall Equation	
Net Ionic Equation	

18. $\text{CaCO}_3\text{(s)} + \text{HCl(aq)}$

Observations	
Overall Equation	
Net Ionic Equation	

19. $\text{NaHCO}_3\text{(aq)} + \text{HCl(aq)}$

Observations	
Overall Equation	
Net Ionic Equation	

20. $\text{Na}_2\text{CO}_3\text{(aq)} + \text{HCl(aq)}$

Observations	
Overall Equation	
Net Ionic Equation	

On this page you perform four additional reactions using the reagents available. Record your observations and write in the equations for any reactions that occur. You may NOT repeat any of the previous reactions, and you may NOT use the same reagents in more than two of the reactions.

21. _____

Observations	
Overall Equation	
Net Ionic Equation	

22. _____

Observations	
Overall Equation	
Net Ionic Equation	

23. _____

Observations	
Overall Equation	
Net Ionic Equation	

24. _____

Observations	
Overall Equation	
Net Ionic Equation	

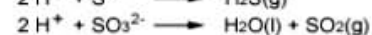
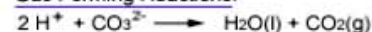
SOLUBLE COMPOUNDS		EXCEPTIONS	
Almost all salts of Na^+ , K^+ , NH_4^+			
Salts of nitrate, NO_3^- chlorate, ClO_3^- perchlorate, ClO_4^- acetate, CH_3CO_2^-			
INSOLUBLE COMPOUNDS		EXCEPTIONS	
Most salts of carbonate, CO_3^{2-} phosphate, PO_4^{3-} oxalate, $\text{C}_2\text{O}_4^{2-}$ chromate, CrO_4^{2-}		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	

Solubility Rules

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-}
$\text{H}_2\text{C}_2\text{O}_4$	$\text{C}_2\text{O}_4^{2-}$
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:



M = a metal atom

Strong Electrolytes:
Soluble ionic compounds
Strong acids and strong bases

Determining Net Ionic Equations

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

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	$\text{Cr}_2\text{O}_7^{2-}$	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)		

Spectroscopic Aspirin Analysis

Objective:

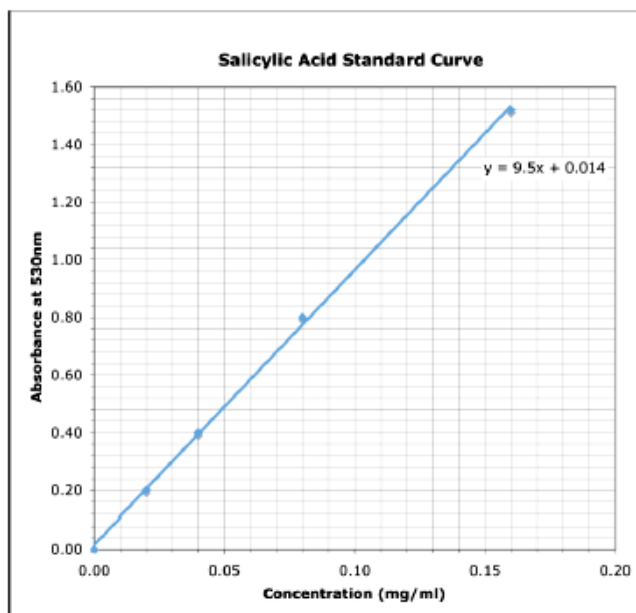
To determine how much active ingredient, acetylsalicylic acid (ASA), is contained in a typical tablet using visible spectroscopy.

Hazards:

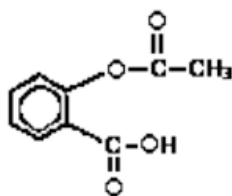
Sodium hydroxide is very corrosive. If you get it on your skin it will feel slippery; rinse with lots of water.

Introduction:

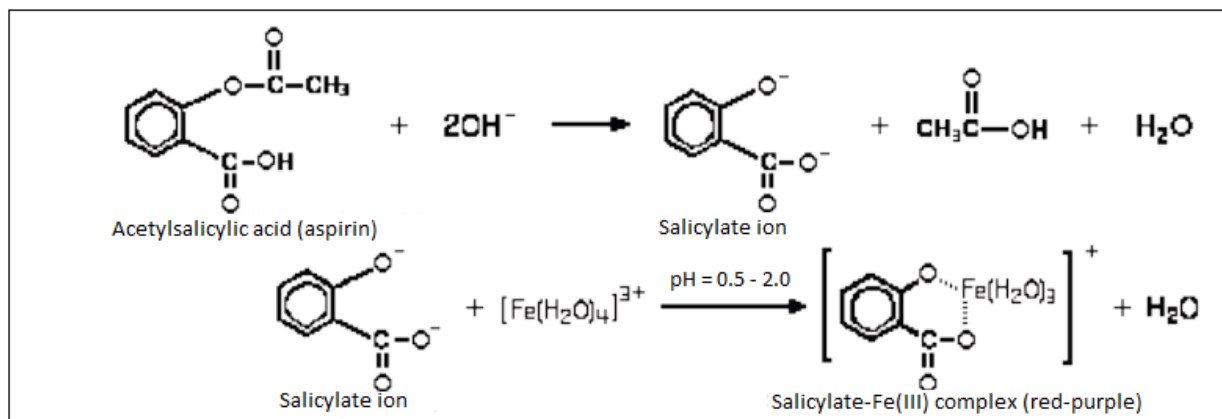
A spectrophotometer is an instrument that can *quantitatively* measure how much light of a certain wavelength is absorbed by a solution. A solution with a high concentration will absorb more light. This relationship can be exploited to determine how much of a substance is in a solution of an unknown concentration via comparison with solutions of known concentrations. The absorbance data from the solutions of known concentrations are used to produce a “**standard curve.**” A standard curve is a graph of the concentration versus the absorbance at a particular wavelength. The relation (i.e. the slope of the line of best fit) between these two quantities can then be used to determine the unknown concentration.



The active ingredient in aspirin is acetylsalicylic acid, or ASA. ASA appears colorless in aqueous solution, but a simple chemical reaction will convert ASA into a complex ion that appears red and absorbs light of 530nm. Since the intensity of the color is directly related to the concentration of the aspirin present, spectrophotometric analysis can be used.



Acetylsalicylic acid (aspirin)



Production of Salicylate-Fe(III) Complex

To produce the colored complex, NaOH will be used to break the bond between the acetyl group and salicylic acid. This leaves the salicylate²⁻ ion, which will form a complex with Fe³⁺ ions. The salicylate-Fe(III) complex is a dark purple-red color that has a maximum absorbance at the wavelength around 525nm (this means the solution absorbs yellow-green light). To determine the amount of salicylate in the aspirin tablet, the absorbance of the solution produced from the aspirin tablet will be compared to the absorbance of “standard” salicylate-Fe(III) solutions.

Procedure:

Part I – Preparing the Aspirin Samples

1. Weigh one aspirin tablet to the nearest 0.01g. Record the mass on your Data Sheet. Place the tablet in a 125mL Erlenmeyer flask
2. Measure 10mL of 1.0M NaOH solution in a clean, dry graduated cylinder. Add the NaOH to the flask containing the aspirin tablet.
3. Heat the mixture to a mild boil for five minutes on a hot plate to hydrolyze the ASA. Be careful to avoid splattering and do not let the solution dry up to prevent loss of contents. Rinse the inside walls of the flask with a small amount of distilled water to ensure complete chemical reaction of the ASA.
4. Quantitatively transfer the solution to a 250ml volumetric flask using a funnel. Thoroughly rinse the flask and funnel with distilled water so that the rinse water flows into the volumetric flask. Add distilled water to the solution in the flask until the bottom of the meniscus touches the index mark of the flask neck. Stopper the flask. While firmly holding the stopper, invert the flask 10 times to thoroughly mix the solution.

- a. Note – The aspirin may have a milky appearance due to starch fillers in the aspirin tablet. Some buffering agents like aluminum hydroxide will not dissolve completely in base. If your solution is cloudy, allow the precipitate to settle to the bottom of the flask. When you take a sample of your solution for analysis, use your pipette to remove solution from the top portion of the liquid so that you will not draw any precipitate into your pipette.
5. Label two test tubes “A” and “B.” Using a 10ml pipette, measure 9ml of FeCl_3 solution and place into tube A. Measure 9.5ml of FeCl_3 solution and place into tube B.
6. Switch to a 1ml pipette. Measure 1ml of your aspirin solution and place into tube A. Mix thoroughly by drawing the mixture up into the pipette and squirting it back into the tube several times.
7. Measure 0.5ml of your aspirin solution and place into tube B. Once again, mix thoroughly as described above. You now have two “unknown” samples; Sample A is TWICE as concentrated as Sample B.

Part 2 – Preparing the Salicylic Acid Standard

1. Label five test tubes 1-5. Take 6ml of FeCl_3 solution and place into tube #1. This is your reference solution. It will be used to define 0% absorbance.
2. Using a 10ml pipette, carefully measure 9ml of FeCl_3 solution and place into tube #2. Place 5ml of FeCl_3 solution into each of the remaining tubes (#3, #4, and #5).
3. Using the 1ml pipette, carefully take 1ml of the ASA standard solution and place into tube #2.
4. Switch to a 5ml pipette. In order to thoroughly mix the solution in tube #2, draw the mixture up into the pipette and squirt it back in several times.
5. Take 5ml of the solution #2 and transfer to tube #3; mix thoroughly.
6. Take 5ml of the solution #3 and transfer to tube #4; mix thoroughly.
7. Take 5ml of the solution #4 and transfer to tube #5; mix thoroughly.

Part3 – Spectroscopic Analysis of Samples

1. Obtain 7 cuvettes and caps from the front desk. Label 5 cuvettes at the top 1-5, and two additional A and B. Make sure they are clean and dry before using. Fill each at least 2/3 of the way full, cap, and clean the outside with a Kimwipe to remove dust and fingerprints. If there are bubbles on the inside of your cuvettes, gently tap on the outside to remove the bubbles.
2. Follow the instructions at the colorimeter station to measure the absorbance of each sample at 525nm. Record all of your measurements on your Data Sheet as well as on the Report Form.
3. Analyze your data as described on the Report Form.

Report:

You will NOT have a separate write-up for this lab. You will be handing in the Report Form as a lab report.

Spectroscopic Aspirin Analysis Data Sheet

On this sheet, write all observations, data, and procedural changes that occur in the lab. Observations should include any descriptions of materials and chemical/physical changes. Data should include all weighed masses, any recorded temperature, times, pH values, calculations, etc.

Spectroscopic Aspirin Analysis Report Form

Name: _____

Date _____

Mass of Aspirin Tablet = _____ g = _____ mg

Table 1 – Standard Curve Data (knowns)			
Tube	Contents	Concentration (mg/ml)	Absorbance at 525nm
1	3ml FeCl ₃ Solution	0.00	
2	1ml ASA Standard 9ml FeCl ₃ Solution	0.176	
3	5ml Tube #2 5ml FeCl ₃ Solution	0.088	
4	5ml Tube #3 5ml FeCl ₃ Solution	0.044	
5	5ml Tube #4 5ml FeCl ₃ Solution	0.022	

Table 2 – Aspirin Sample Data (unknowns)				
Tube	Contents	Dilution Factor	Concentration* (mg/ml)	Absorbance at 525nm
A	1ml Aspirin Solution 9ml FeCl ₃ Solution	10		
B	0.5ml Aspirin Solution 9.5ml FeCl ₃ Solution	20		

*The concentration of colored product in tubes A and B will be determined by comparing the absorbance with the absorbance of the "standard," or known samples (tubes 1-5), using a Standard Curve. Note that Tube A is TWICE as concentrated as Tube B, so it should absorb twice as much light.

Data Analysis – Making the Standard Curve Graph

In order to determine the amount of ASA in the aspirin tablet, you will have to compare the absorbance of your aspirin samples to that of the known concentrations of ASA (tubes 1-5). Use EXCEL or another graphing program to construct a plot of the absorbance (y-axis) versus concentration (x-axis) for tubes 1-5 only (DO NOT include tubes A and B in this plot!).

Fit the line of best fit for your data by Right-Clicking on the data points on the graph and choosing Add Trendline. It should be a linear line that goes through the 0,0 point on the graph. Be sure to display the equation of the line and the R² value.

This is your standard curve. It tells you the relationship between the absorbance of the salicylic acid solution and its concentration. Based on the equation for your standard curve, determine the concentration value for samples A and B. Remember, the concentration is represented by the x-value, so you must solve for x. **PRINT OFF AND ATTACH YOUR GRAPH TO THIS REPORT FORM.**

Calculations

To determine the amount of ASA in an aspirin tablet:

$$(\text{concentration in mg/ml})(\text{dilution factor})(250\text{ml}) = \underline{\hspace{2cm}} \text{mg in original tablet}$$

Sample A (dilution factor = 10):

Sample B (dilution factor = 20):

Average of Sample A and Sample B:

Questions

1. Why did you use the FeCl_3 solution as a reference solution or “blank”? Would water have worked just as well? Explain your reasoning.

2. Compare your results for tubes A and B. Do they agree (i.e. did both samples give you the same answer for the amount of ASA in an aspirin tablet)? Explain how **precise** your data is, be quantitative. If the two values differ, provide a possible explanation.

3. Compare your value for the ASA content of an aspirin table with the advertised value (the #mg per tablet listed on the bottle = 325mg). How close were your results to the actual value? Assume that the advertised value is correct; how **accurate** were your results, be quantitative and calculate the % error?

$$\% \text{ error} = \left| \frac{325 \text{ mg} - \text{Your value (mg)}}{325 \text{ mg}} \times 100\% \right|$$

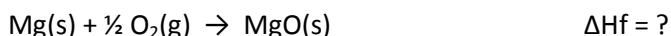
Thermochemistry: The enthalpy of formation

Objective:

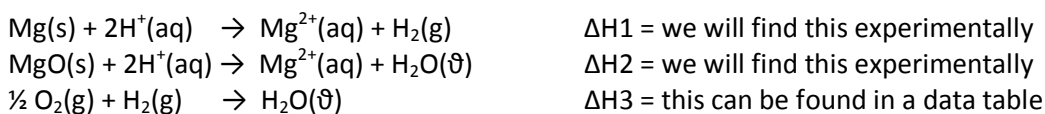
The purpose of Parts Ia and Ib of this experiment is to determine the enthalpy of formation of a compound, magnesium oxide, MgO. The purpose of Part II of this experiment is to observe the heat evolved from neutralizing acids and bases of varying strengths.

Introduction:

The formation reaction for Magnesium oxide is:



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

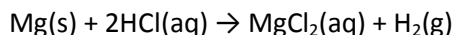


Once the three values, ΔH_1 , ΔH_2 and ΔH_3 , are obtained, Hess's Law can be used to combine those values such that the desired heat of formation can be calculated.

Procedure:

PART Ia - 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.



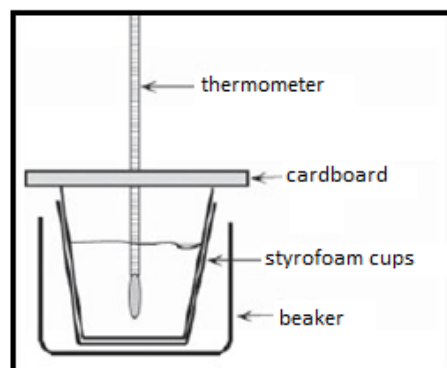
1. Set up your coffee-cup calorimeter and support it in a ring stand or beaker as illustrated.



(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, say about 0.2 g, about 0.4 g, and about 0.5 g. Record the masses in your lab notebook.

3. Place one of the magnesium samples in a clean, dry coffee-cup calorimeter.
4. Using a graduated cylinder, measure out as accurately as possible 100.mL of 1.0 M HCl. Measure the temperature of the HCl solution and record this initial temperature in your lab notebook. Replace the thermometer in the calorimeter setup.
5. Add the HCl solution to the calorimeter and swirl gently but steadily. At 30 second intervals record the temperature (to the nearest 0.5 °C) until the temperature has held constant or decreased for three consecutive readings.
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 Ib - Determining ΔH_2 : Heat of Reaction of Magnesium Oxide with Hydrochloric Acid

1. Set up the calorimeter as in PART I. Make sure it is clean and dry.
2. Weigh out about 0.7 g of MgO to the nearest 0.001 g and place the powder in the calorimeter.
3. Repeat steps (d) and (e) as in PART I above with another sample of magnesium oxide.

Calculations for the Report for Parts Ia and Ib:

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 we will assume that they have a specific heat capacity of 4.18 J/g·°C and a density of 1.00 g/mL.

1. Use the equation,

$$\# J = \text{Specific heat capacity} \times \text{mass (g)} \times \Delta T$$

to determine the quantity of heat released. Then, using this and the number of moles of compound reacting, determine the value of ΔH for each reaction.

$$\Delta H = \frac{\#J}{\# \text{ moles}}$$

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

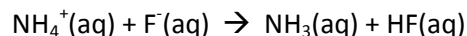
2. Report the value of ΔH for each reaction.
3. Use the three reactions performed in Part 1 to verify if the amount of heat released is proportional to the number of moles of compound reacting. Report on whether this is true.
4. Use the ΔH values determined experimentally as well as that for formation of H₂O 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).**

Part II - Exploring the heat of neutralization of acids and bases.

Introduction:

All acid-base reactions involve the same basic step: the transfer of an H⁺ ion (which is just a proton) from the acid to the base. An example is the reaction between NH₄⁺ and F⁻ ions.



In this case the ammonium ion, NH₄⁺, is the acid and fluoride ion (F⁻) is the base.

In this experiment you will perform reactions between the following two acids and bases:

<u>Acids</u>	<u>Bases</u>
Hydrochloric (HCl)	Ammonia (NH ₃)
Acetic (CH ₃ COOH)	Sodium hydroxide (NaOH)

You will react each acid with each base, so you will perform four reactions.

Procedure:

1. Similarly to Part I, pour 25 mL of the acid (measured with a graduated cylinder) into the Styrofoam cup and measure its temperature.
2. Add 25 mL of the base solution, place the lid with thermometer on top, stir, and measure the temperature change.

Calculations for the Report for Part II:

1. Calculate the enthalpy change for each reaction in terms of kJ/mol.
2. Report the four enthalpy changes you determined.
3. Make a statement explaining the trends you see in terms of the strengths of the acids and bases used.
4. Write Net Ionic equations for each of the reactions after the Conclusion section of the report.

Report:

Complete 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.

Follow the guidelines for the Calculations for the Report for Parts Ia and Ib as well as Part II. Show all of your work for the calculations on the attached Thermochemistry Calculations Sheet.

Attach the Thermochemistry Calculations Sheet to your Report.

Name _____

Thermochemistry Calculations Sheet:Convert all ΔH answers from J/mol into KJ/mol

Reaction 1a		$\Delta H_1 =$
Reaction 1b		$\Delta H_2 =$
	$\frac{1}{2} \text{O}_{2(g)} + \text{H}_{2(g)} \rightarrow \text{H}_2\text{O}_{(l)}$	$\Delta H_3 = -285.5 \text{ KJ/mol}$
Net Reaction		$\Delta H_{\text{MgO}} =$

Part 1a:Calculate ΔH_1 for $0.2\text{g Mg}_{(s)} + 2\text{H}^+$ Calculate ΔH_1 for $0.4\text{g Mg}_{(s)} + 2\text{H}^+$ Calculate ΔH_1 for $0.5\text{g Mg}_{(s)} + 2\text{H}^+$ Calculate Average ΔH_1 _____

Part 1b:

Calculate ΔH_2 for $0.7\text{g MgO}_{(s)} + 2\text{H}^+$

Calculate ΔH_2 for $0.7\text{g MgO}_{(s)} + 2\text{H}^+$

Calculate Average ΔH_2 _____

- Now plug these values into the table above, rearrange the equations, and solve for the net reaction for the heat of formation of $\text{MgO}_{(s)}$

Part 2:

When doing the math for this section, mass will be 50g, and you need to solve for the moles by using the molarity and volume of the acid. Balance the Overall Equations.

Overall Reaction: _____

Calculate ΔH

Overall Reaction: _____

Calculate ΔH

Overall Reaction: _____

Calculate ΔH

Overall Reaction: _____

Calculate ΔH

Thermochemistry Data Sheet

On this sheet, write all observations, data, and procedural changes that occur in the lab. Observations should include any descriptions of materials and chemical/physical changes. Data should include all weighed masses, any recorded temperature, times, pH values, calculations, etc.

Copper Complex Analysis: Part I

Objective:

In this experiment you will synthesize a compound by adding NH_3 to a concentrated aqueous solution of copper sulfate. The blue CuSO_4 solution will turn a still deeper blue and a mass of small deep blue-to-violet crystals will form as ethyl alcohol is added.

After making the compound, you will do three separate analyses to determine the amount of Cu^{2+} , SO_4^{2-} and NH_3 in your product. The tests will include gravimetric, volumetric, and spectrophotometric experiments, all of which you performed in previous experiments.

You will then use your results to propose a formula for the compound. The general formula for the compound is: $\text{Cu}_x(\text{NH}_3)_y(\text{SO}_4)_z \cdot a\text{H}_2\text{O}$, where x, y, z, and a are integers which you will determine from the analysis of your product.

Hazards:

- Work in the hood when using concentrated NH_3 .
- Acetone is flammable

Introduction:

Four principal species are present initially in the reaction mixture: copper (II) ions [actually $\text{Cu}(\text{H}_2\text{O})_6^{2+}$ ions], ammonia molecules (NH_3), sulfate ions (SO_4^{2-}), and water. We will assume that the product forms as a result of the reaction of two or more of these species. Ethanol is also present, but it is an indirect participant in the reaction. In aqueous solutions ethanol, this is miscible with water but of lower dielectric constant decreases the solubility of ionic compounds. The marked color change that occurs in the reaction is an important clue to the nature of the product.

In the following week, the product will be analyzed for copper (II) ions, sulfate ions and ammonia molecules. Water content will be determined by subtraction, once the proportions of the other species are known. 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. With careful attention to detail and techniques one can obtain excellent results for each part of the analysis.

Procedure:**A. Synthesis of the Copper Compound**

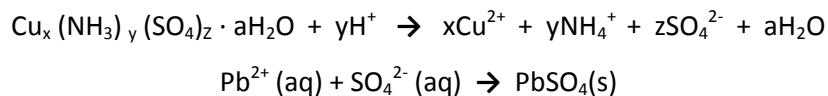
1. Weigh out 10.0 grams of copper sulfate pentahydrate, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ and place the crystals in a 250mL beaker. Record the exact mass in your lab notebook.
2. Add 20-15mL of water to the solid and then in a hood add 20mL of 15M NH_3 (concentrated ammonia). Stir until the crystals are completely dissolved.
3. Over a period of 1 minute, slowly add 20mL of 95% ethanol to the solution, stir, and cool to room temperature.
4. While the mixture is cooling, combine 15mL of concentrated ammonia and 15mL 95% ethanol. Cover all ammonia-containing solutions with watch glasses (to prevent the fumes of NH_3 from saturating the lab) and bring them back to your station.
5. Set up a vacuum filtration apparatus, moisten the filter paper and turn on the aspirator. Carefully filter the slurry of crystals that has formed in the copper- containing solution and suck off all the solution.

If a significant amount of crystalline product remains in the beaker, you should use the filtrate from the filter flask to wash the crystals on the filter paper. To prevent backup of tap water into the filter flask, which would render the filtrate useless as a wash, pull the hose off the aspirator while the water is still running. Remove the filter funnel and pour the filtrate from the filter flask back into the beaker that contains the crystals. Reassemble the filter apparatus and collect the remaining product on the filter paper.

- Turn off the aspirator and carefully pour 10mL of the ammonia-ethanol solution onto the crystals. Break up all lumps of solid to permit the liquid to penetrate the mass completely and then turn on the aspirator to suck off the liquid. Repeat the washing procedure twice. Next, wash twice with 10mL portions of 95% ethanol and finally with two 10mL portions of acetone, breaking up the mass of crystals with a spatula in each step before turning on the aspirator. (At this point your crystals should appear nearly "dry", that is, not moistened with liquid to any great extent. If you feel they could be dried more, add additional amounts of acetone as described above.) To remove the last traces of moisture and other solvents from your solid, draw air through the crystals for at least 5 minutes, use your spatula to break up any remaining lumps.
6. Put the crystals in a large test tube or small beaker, lightly cover, and leave them in your locker to dry thoroughly until the next laboratory period. You will record the mass of your crystals next week. You will not use your crystals for the analysis of the sulfate ion (SO_4^{2-}) though; you will use a premade sample of the copper compound to analyze the sulfate ion.

B. 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 (not the one you made) 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:



1. Weigh about 0.9-1.1 g of the copper compound to the nearest 0.001 g.
2. Dissolve the solid in 10mL of 6 M nitric acid (HNO_3).
3. Obtain 5-6mL of 1M lead acetate [$\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2$], and add it dropwise to the acid solution until precipitation is complete.

How to determine when precipitation is complete: First allow all the white precipitate to settle to the bottom of the beaker. Using your eyedropper, allow one drop of lead acetate solution to run down the side of the beaker into the light blue solution. If precipitation of the SO_4^{2-} ion is not yet complete, you will see a white precipitate of PbSO_4 form just as the drop slides down the beaker wall and into the solution. If you do not observe a white solid forming at this point, then precipitation is complete and you can proceed.

4. Once you have determined that the PbSO_4 precipitation is complete, weigh a piece of filter paper to the nearest milligram. Assemble the apparatus 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. Using a stirring rod to guide the flow of liquid, 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. Since you must do a duplicate determination, you may wish to start the second one while the first one is filtering.
6. After most of the liquid has collected from the filtration, check it once again for completeness of precipitation by adding a few drops of lead acetate. If a white precipitate forms (i.e., the solution becomes cloudy), add another milliliter of lead acetate solution and refilter.
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. Rinse the precipitate with a small portion (about 10mL) of acetone.
9. When the liquid from the last washing has drained out remove the filter paper (be careful not to tear it!) and place it on a watchglass to dry until next lab.
10. Repeat this procedure to produce a second PbSO_4 sample. Somehow label which filter paper is trial 1 and which is trial 2.

Next Week:

You will do the analysis of the PbSO_4 you synthesized, and then complete the Volumetric Analysis for Ammonia and the Spectrophotometric Analysis for Copper.

Copper Complex Analysis Part I Data Sheet

On this sheet, write all observations, data, and procedural changes that occur in the lab. Observations should include any descriptions of materials and chemical/physical changes. Data should include all weighed masses, any recorded temperature, times, pH values, calculations, etc.

Copper Complex Analysis: Part II

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 lab report.

Procedure:

NOTES: Before doing any of the three analyses, make sure that your compound is completely dry. Each analysis will enable you to determine the mass % of one component of the copper compound, which can then be used to determine the overall formula.

A. Percent Yield and Mass Percent Calculations

Complete this step before doing any compound analysis.

1. Weigh your entire copper compound to the nearest 0.001 g and record the mass on your Data Sheet and on your report form (as the "Mass of Product Isolated").
2. Weigh your $PbSO_4$ precipitate and filter paper to the nearest milligram, record the mass on your Data Sheet. Calculate the mass percent in your notebooks and on the report form, use the example below as a guide.

EXAMPLE OF MASS PERCENT CALCULATION for SO_4^{2-}

Mass of copper compound used in gravimetric analysis = 1.089 g

Mass of $PbSO_4$ isolated = 1.252 g

- Calculate the moles of $PbSO_4$

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

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

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

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

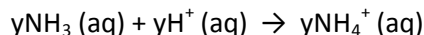
$$0.004129 \text{ mol } SO_4^{2-} \times \frac{96.06 \text{ g } SO_4^{2-}}{1 \text{ mol } SO_4^{2-}} = 0.396594 \text{ g } SO_4^{2-}$$

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

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

B. Volumetric Analysis for Ammonia, NH₃

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



where H⁺ is from a standardized solution of strong acid, HCl.

1. Weigh two samples of your compound to the nearest 0.001 g and record their masses. Dissolve each in about 30mL of water in an Erlenmeyer flask. (Do not allow the solutions to stand very long before titrating.)
2. 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 on your Data Sheet, along with the beginning and final burette readings.

Numerous color changes will occur as the acid is added. The solution starts out a deep blue. However, as acid is added the color changes to a blue-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.

3. 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.

EXAMPLE OF MASS PERCENT CALCULATION for NH₃

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

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

- Use the stoichiometry from equation (3) to determine the moles of NH₃

$$0.015519\text{ mol HCl} \times \frac{1\text{ mol NH}_3}{1\text{ mol HCl}} = 0.015519\text{ mol NH}_3$$

- Convert the moles of NH₃ to grams

$$0.015519\text{ mol NH}_3 \times \frac{17.03\text{ g NH}_3}{1\text{ mol NH}_3} = 0.264284\text{ g NH}_3$$

- Use the grams of NH₃ and the mass of the copper compound to determine the mass % of NH₃

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

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

- Clean and dry six 18 x 150 mm test tubes (the smaller size in your drawer), and label them 1 through 6. The first four of these tubes will be used to make up the reference solutions that you will use to produce a **standard curve**.
- Prepare your standards and samples of the unknown compound as described in the table below. Record the concentration of the standard Cu^{2+} solution.

Tube #	Contents
1	About 10ml of 1M HNO_3
2	4.0ml standard Cu^{2+} solution and 6.0ml 1M HNO_3 . Mix well.
3	7.0ml standard Cu^{2+} solution and 3.0ml 1M HNO_3 . Mix well.
4	10.0ml standard Cu^{2+} solution
5	Weigh ~0.2g of Copper Compound to the nearest 0.001g. Add exactly 10.0ml 1M HNO_3 . Mix well.
6	Weigh ~0.3g of Copper Compound to the nearest 0.001g. Add exactly 10.0ml 1M HNO_3 . Mix well.

- Take your samples and 7 plastic cuvettes (available on the front bench) over to one of the colorimeters.
- Follow the instructions at the colorimeter station to measure the absorbance of each sample at 630 nm (Red LED). Use the contents of tube 1 as the reference sample. This means that when calibrating the colorimeter, you should use the contents of tube 1 to fill two cuvettes. Record your measurements on your Data Sheet.

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

Mass of copper compound used in spectrophotometric analysis = 0.215 g
 Concentration of Cu^{2+} standard = 0.16 M

Tube	Absorbance @ 630 nm
1	0.01
2	0.31
3	0.54
4	0.77
5	0.39
6	0.56

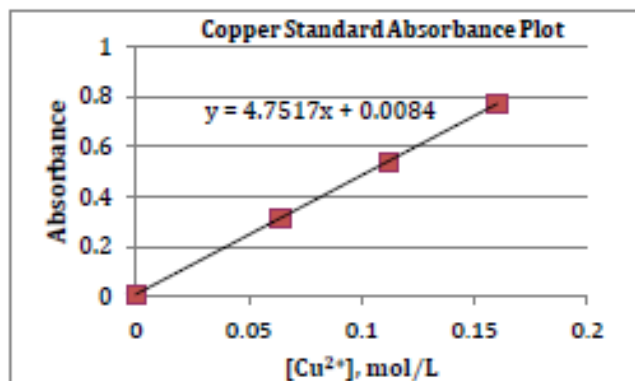
- Calculate the concentration of Cu^{2+} in tubes 1-4. To do this, multiply the concentration of the standard Cu^{2+} solution by the dilution factor for the tube. For example, the concentration in tube 2 in this case would be:

$$\frac{0.16 \text{ mol}}{1 \text{ L}} \text{Cu}^{2+} \times \frac{4 \text{ mL}}{10 \text{ mL}} = 0.064 \frac{\text{mol}}{\text{L}} \text{Cu}^{2+}$$

dilution factor

(Note that the numerator of the dilution factor is the volume of Cu^{2+} standard solution in the tube and the denominator is the total volume in the tube. Thus, the concentration of Cu^{2+} in tube 1 is 0 M.)

- Prepare a plot of Absorbance vs. Concentration using the data from tubes 1-4. Include a line of best fit, or trendline (see instructions for graphing absorbance data in the aspirin lab). This is your "standard curve". Make sure to include the graph in your report.



- Use the equation for the best fit line to determine the Cu^{2+} concentration in tubes 5 and 6. In this example, the absorbance (or y value) for Tube #5 is 0.39, so the concentration of Cu^{2+} is:

$$[\text{Cu}^{2+}] = \frac{0.39 - 0.0084}{4.7517} = \frac{0.080317 \text{ mol}}{\text{L}}$$

- Calculate the moles of Cu^{2+} in sample #5

$$\frac{0.01 \text{ L}}{\text{total volume in tube \#5}} \times \frac{0.080317 \text{ mol}}{1 \text{ L}} = 0.000803 \text{ mol Cu}^{2+}$$

concentration in tube \#5

- Convert the moles of Cu^{2+} to grams

$$0.000803 \text{ mol Cu}^{2+} \times \frac{63.55 \text{ g Cu}^{2+}}{1 \text{ mol Cu}^{2+}} = 0.051042 \text{ g Cu}^{2+}$$

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

$$\frac{0.051042 \text{ g}}{0.215 \text{ g}} \times 100\% = 23.740\%$$

PUTTING IT ALL TOGETHER

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-}	$23.74 + 25.834 +$ $36.418 = 85.992$	XXXXXXXXXX		
H_2O = $100 - (\text{total of } \text{Cu}^{2+},$ $\text{NH}_3, \text{ and } \text{SO}_4^{2-})$	$100 - 85.992 =$ 14.008			$a=$

Calculation of "Moles per 100g" for Cu^{2+}

$$23.74\text{g} \times \frac{1\text{mol Cu}^{2+}}{63.55\text{g}} = 0.3736$$

To get the "Mole Ratio" for each component, divide by the # moles of Cu^{2+} in 100g. In the above example, the moles/100g for NH_3 , SO_4^{2-} , and H_2O would each be divided by 0.374.

Copper Complex Analysis Part II Data Sheet

On this sheet, write all observations, data, and procedural changes that occur in the lab. Observations should include any descriptions of materials and chemical/physical changes. Data should include all weighed masses, any recorded temperature, times, pH values, calculations, etc.

Copper Compound Analysis Report Form

Name _____ Section _____

Grade ____/20

SUMMARY OF RESULTS AND CONCLUSIONS

Data for Cu^{2+} mass % determination	Concentration of Cu^{2+} standard		
	Standard #1 Absorbance		
	Standard #2 Absorbance		
	Standard #3 Absorbance		
	Standard #4 Absorbance		
	Mass of samples used in spectrophotometric analysis		
	Absorbance of samples		
	AVERAGE MASS % OF Cu^{2+}		
Data for NH_3 mass % determination	Concentration of HCl		
	Mass of sample used in volumetric analysis		
	Volume of HCl used in titration		
	AVERAGE MASS % OF NH_3		
Data for SO_4 mass % determination	Mass of samples used in gravimetric analysis		
	Mass of PbSO_4		
	AVERAGE MASS % OF SO_4		
Data for % Yield Calculation	Mass of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ used in synthesis		
	Mass of <i>Your Product Isolated</i>		
FORMULA OF COMPOUND			
THEORETICAL YIELD			
% YIELD			

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:

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:

SPECTROPHOTOMETRIC ANALYSIS FOR COPPER

Sample	[Cu ²⁺]	Absorbance @ 630nm (Red LED)	
Standard 1	0.0 M		Mass % Copper
Standard 2			
Standard 3			
Standard 4			
Sample 5			
Sample 6			
Average Mass %			

Make a standard curve using Excel or some other graphing program (refer to the Aspirin lab instructions). Remember, x-axis should be the [Cu²⁺] and the y-axis should be absorbance. **Attach your graph to this report.**

Write the equation for the trendline, standard curve, below (it should be in $y = m x + b$ form):

Sample calculation of [Cu²⁺] (use the absorbance value for Sample A and the above equation):

Sample of mass % calculation:

**CALCULATION OF THE FORMULA OF THE COORDINATION COMPOUND
AND THE PERCENT YIELD**

	Weight Percent = g in 100g	Moles per 100g	Mole Ratio	
Cu ²⁺				x=
NH ₃				y=
SO ₄ ²⁻				z=
Total Mass of Cu ²⁺ , NH ₃ , and SO ₄ ²⁻				
H ₂ O = 100 - (total of Cu ²⁺ , NH ₃ , and SO ₄ ²⁻)				a=

General formula of compound = $\text{Cu}_x(\text{NH}_3)_y(\text{SO}_4)_z \cdot a\text{H}_2\text{O}$

Formula of compound (your experimental results) = _____

Molar mass of compound (find using above formula) = _____

Mass of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ used in synthesis = _____

Calculation of Theoretical yield:

Mass of Product Isolated = _____

Calculation of % Yield:

Periodic Properties of the Elements

Objective:

The purpose of this experiment is to explore the chemistry of a few of the elements, both in the laboratory and by computer. You will look for trends in the chemical and physical properties of the elements as a function of the position of the elements in a periodic group.

Hazards:

Sodium and Lithium are extremely flammable in water – be careful!
Hexane is extremely flammable.

Introduction:

1. Follow the experimental procedure detailed in the laboratory write-up.
2. Record your observations in the Report Form for this laboratory for the reactions observed. In this experiment, some reactions occur immediately, some take longer, some never occur. (The element being investigated is always the limiting reagent, if it reacts, all of it should be consumed.) Note any and all reactions that may occur. Especially record similarities and differences between different elements within the same group and between elements of different groups. From your observations, try to determine the form and formula of the resulting products.
3. From your observations, complete the Report Form packet and hand it in at the end of lab.
4. Use distilled or distilled water in all experiments calling for water. Record all observations on the accompanying data sheets as you do the experiment. Write chemical equations for all reactions observed; if no reaction is observed, write n.r. in the appropriate space.

Procedure:

A. The Group IA Elements: Alkali Metals–Lithium and Sodium

1. Using a clean, dry watchglass, get a small piece, about 1/8 in. diameter (about 0.03 g) of metallic lithium and sodium under kerosene or oil. Handle these pieces with dry forceps or tongs. Remove the excess kerosene or oil from the metal by gently scraping the sides with the knife available to cut the metal.
2. Cut the piece of sodium metal with the end of a spatula and simultaneously observe what happens as the freshly cut surface is exposed to the atmosphere.
3. Drop the small piece of sodium metal into 50mL of distilled water in a 250mL beaker, simultaneously covering the beaker with a wire gauze. Keep the beaker at arm's length. Test the resulting solution with litmus paper.
4. Carry out the same reaction with lithium metal. Record your observations.

3 Li 6.941
11 Na 22.998
19 K 39.0983
37 Rb 85.4678
55 Cs 132.905

The Group IA alkali metal elements

B. The Group 2A Elements: Alkaline Earth Metals—Magnesium and Calcium

1. Grasp a small piece of magnesium ribbon with crucible tongs and, while holding it at arm's length, insert it in the burner flame. Do not look directly into the flame. Record your observations and write the balanced equation for the reaction.
2. Repeat the test with a small piece of calcium. Record the observations.
3. Scratch a small piece of magnesium ribbon with a spatula. Make up a test tube of 5mL of water and a few drops of phenolphthalein indicator. Place your magnesium in the test tube. Record your results.
4. Repeat this test with water and a small piece of calcium. (It may be necessary to break the calcium into small pieces.) Record the observations and note the difference in behavior of these elements toward water as compared to the metals of Group 1A. Write equations for the reactions.
5. Place 5mL of 6 M HCl in each of two test tubes. Carefully drop a piece of magnesium into one test tube and a piece of calcium into the other. Place an inverted test tube over one of the test tubes to collect a sample of the gas being evolved. Immediately put the inverted test tube over an open flame. Record the observation.

4	Be	9.0122
12	Mg	24.305
20	Ca	40.078
38	Sr	87.62
56	Ba	137.327

Most of the Group 2A or alkaline earth metals

5	B	10.81	6	C	12.011
13	Al	26.9815	14	Si	28.0855
31	Ga	69.723	32	Ge	72.61
49	In	114.82	50	Sn	118.710
81	Tl	204.383	82	Pb	207.2

Groups 3A and 4A of the periodic table.

C. The Group 3A Elements—Aluminum

1. Grasp a small piece of aluminum wire with crucible tongs and, while holding it at arm's length, insert it in the burner flames. Record the observations.
2. Place about 0.25 g of granular aluminum in a test tube containing water and another sample of aluminum in a 150mL beaker containing 5mL of 6M HCl. Record the observations. Write a balanced equation for any reaction you may observe.

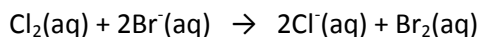
D. The Group 4A Elements—Carbon

Place about 0.25 g of powdered carbon in a test tube containing water and another sample of carbon in a test tube containing 5mL of 6 M HCl. Tap the bottom of each tube gently with a finger. Wait 5 minutes, and then tap each tube again. Record your observations.

E. The Group 7A Elements: the Halogens—Chlorine, Bromine, and Iodine

Here you will place a small quantity of aqueous solutions of each of the halogens (Cl₂, Br₂, and I₂) in a small test tube. On adding hexane, an organic liquid that does not mix with water, you will see two layers in the test tube.

After observing the color of the halogen dissolved in hexane, the next step is to mix a halogen with the salt of another halogen and then add hexane. In some cases, the halogen will oxidize the halide ion in a reaction such as



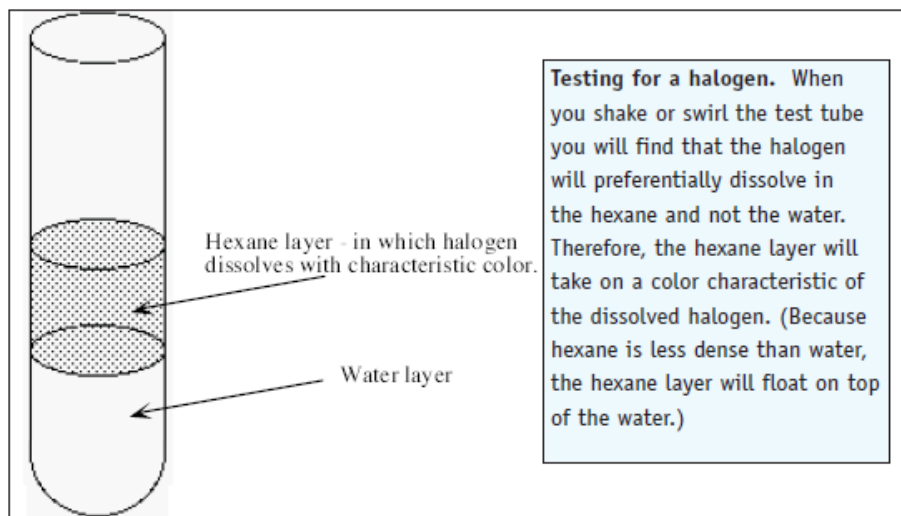
When this particular reaction occurs, the bromine released in the reaction dissolves in the hexane layer, and you observe the characteristic color of bromine in the hexane.

Part E-1: Color of Halogens in Hexane

The purpose of this part of the experiment is to discover the color that each halogen (Cl_2 , Br_2 , and I_2) has in the organic solvent hexane.

1. *Add chlorine water*

Place about 2mL of chlorine water (chlorine dissolved in water), bromine water, and iodine water into separate test tubes. Record the appearance of each one.



9	F	18.9984
17	Cl	35.4527
35	Br	79.904
53	I	126.9045
85	At	(210)

The Group 7A elements or the halogens

2. *Add hexane*

To each test tube, add about 2mL of hexane. Mix the contents of each test tube by shaking or swirling it carefully. Record your observations, particularly the color of the hexane layer.

Part E-2: Reactions of the Halogens

The purpose of this part of the experiment is to find out which halogen can oxidize which halide ions or ions. If such a reaction occurs, the product halogen will be dissolved in the organic solvent (hexane) and, from the color observed above, you can decide if the reaction occurred.

3. *Reaction of Cl_2 with Br^- and Cl_2 with I^-*

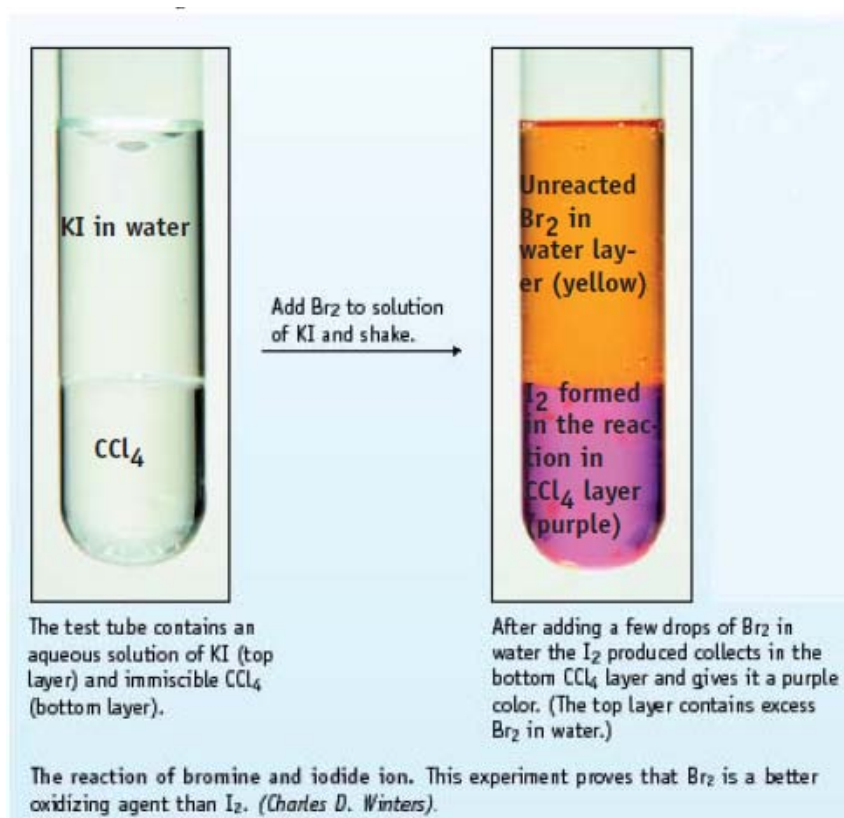
Add a few drops of chlorine water (chlorine dissolved in water) to a clean test tube containing 1mL of 0.2 M KBr and to a second test tube containing 1mL of 0.2 M KI.

4. *Reaction of Br_2 with Cl^- and Br_2 with I^-*

Add a few drops of bromine water (bromine dissolved in water) to another test tube containing 1mL of 0.2 M NaCl and to a test tube containing 1mL of 0.2 M KI.

5. *Reaction of I_2 with Cl^- and I_2 with Br^-*

Add a few drops of iodine water (iodine dissolved in a KI solution) to a different test tube containing 1mL of 0.2 M NaCl and to a second test tube containing 1mL of 0.2 M KBr.



Br₂ can oxidize I⁻ to I₂. The I₂ dissolves preferentially in a non-polar solvent like CCl₄ more readily than in polar water.

6. *Add hexane to each test tube above*

Add 2mL of hexane (C₆H₁₄) to each solution from steps 3 through 5 and mix the contents thoroughly (by shaking or swirling it carefully). Record your observation on the data sheet. In particular look for evidence of reaction by examining the color of the hexane layer.

F. The Transition Elements—Iron, Copper, and Zinc

1. Place 5mL of water in each of three test tubes. Now place a piece of iron (steel wool) in one test tube, copper in a second, and zinc in a third. Tap each tube. Record the observations.
2. Repeat the experiment with 2mL of 6 M HCl and a sample of each metal. Record the observations.
3. Repeat the experiment with 2mL of 6 M nitric acid, HNO₃ and a sample of each metal. Record the observations.

Periodic Properties of the Elements Report Form

Name _____ Section _____

Grade ____/10

OBSERVATIONS, DATA, AND BALANCED EQUATIONS

A. The Group IA Elements: Alkali Metals – Lithium and Sodium

Lithium and Sodium with Oxygen (O₂)

Element	Observations	Balanced Equation
Lithium		
Sodium		

Lithium and Sodium with water; litmus test

Element	Observations	Balanced Equation
Lithium		
Sodium		

B. Group IIA Elements: Magnesium and Calcium

Reactions with O₂: Flame Test

Element	Observations	Balanced Equation
Magnesium		
Calcium		

Reactions with Water using phenolphthalein acid-base indicator

Element	Observations	Balanced Equation
Magnesium		
Calcium		

B. Group IIA Continued: Reactions with Hydrochloric Acid

Element	Observations	Balanced Equation
Magnesium		
Calcium		

C. Group IIIA: Aluminum

Reaction	Observations	Balanced Equation
Al + O ₂ Flame Test		
Al + H ₂ O		
Al + HCl		

D. Group IVA: Carbon

Reaction	Observations	Balanced Equation
C + H ₂ O		
C + HCl		

E. Reactions of Halogens

E1: Colors of Halogens in Solutions

Observations of solutions of the elemental halogens: Cl₂, Br₂, and I₂ in water

Halogens	Observations of Solution Colors
Cl ₂	
Br ₂	
I ₂	

Observations of solutions of the elemental halogens: Cl₂, Br₂, and I₂ in hexane

Halogens	Observations of Solution Colors
Cl ₂	
Br ₂	
I ₂	

E2. Reactions of halogens with halide ions.

Observations of solution colors after mixing halogens in water with salts containing other halide ions.

	NaCl	KBr	KI
Cl ₂	Don't Bother		
Br ₂		Don't Bother	
I ₂			Don't Bother

Mixture	Balanced Equation for Reaction: Write N.R. if no reaction occurs
Cl ₂ and KBr	
Cl ₂ and KI	
Br ₂ and NaCl	
Br ₂ and KI	
I ₂ and NaCl	
I ₂ and KBr	

F. Transition Metals: Iron, Copper, and Zinc

Iron, Copper and Zinc with Water

Reaction	Observations	Balanced Equation
Fe + H ₂ O		
Cu + H ₂ O		
Zn + H ₂ O		

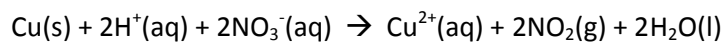
Iron, Copper and Zinc with Hydrochloric Acid

Reaction	Observations	Balanced Equation
Fe + HCl		
Cu + HCl		
Zn + HCl		

Iron, Copper and Zinc with Nitric Acid

Reaction	Observations	Balanced Equation
Fe + HNO ₃		
Cu + HNO ₃		
Zn + HNO ₃		

These reactions are complex. As an example, the net ionic equation for the reaction of copper and nitric acid is:



Questions

Answer the following to explain your observations. Keep in mind the relative position of each element that you investigated in the periodic table. Remember that atoms' uppermost orbital energies generally increase moving from top to bottom of the periodic table, and move down in energy moving from left to right across the periodic table.

1. What trend can you note for the relative reactivities of sodium and lithium and their respective position in the periodic table?

2. Compare, briefly, the similarities and differences in the properties of the Group 2A elements with the Group 1A elements you investigated. Can you rationalize their contrasting behavior? Use the position of the elements in the periodic table and, if possible, the electron configuration for each element.

3. How did the reactivities for the representative elements of Group 3A (Al) and Group 4A (C) compare to each other and to the reactivities of the elements you investigated in Group 1A and 2A. Be specific and give examples to illustrate your findings.

4. Compare the similarities and differences in the properties of the three transition elements you investigated in their combination with water, hydrochloric acid, and nitric acid. Is this behavior consistent with their relative position the periodic table.

5. Halogen Chemistry

a) Which halogen— Cl_2 , Br_2 , or I_2 —is most reactive? How do you conclude this?

b) Which halogen— Cl_2 , Br_2 , or I_2 —is least reactive? How do you conclude this?

c) Is there a trend you can note between the order of reactivity of these Group 7A elements and their position with respect to each other in the periodic table?

d) Is the trend in reactivities similar to any trend noted for the Group 1A or 2A elements you observed.