This experiment was designed and written by Gordon Bain

Purpose of the Experiment

To utilize skills learned in CHEM 103 in the preparation and characterization of a chemical compound.

To demonstrate mastery of fundamental techniques.

To review and apply fundamental concepts taught in the lecture course.

To apply previously separated concepts in the solution of a single problem.

For Your Safety

1. 6 molar hydrochloric acid (HCl) is a concentrated (18% w/w) strong acid.

   CAUTION - this acid will burn skin immediately on contact.

   CAUTION - this acid will fume when heated - **heat only under a fume hood**.

   Wear gloves when handling this chemical.
2. In the event of skin contact with either aqueous hydrochloric acid *or HCl fumes*, rinse the affected area with cold water for 5 minutes.
3. If you should get 6M HCl on your clothing, remove the article of clothing and soak the affected area with cold water. Treat the skin under the affected area as outlined in 2.
4. The surface of the hot-plate will be hot enough to burn skin on contact. Treat thermal burns by rinsing under cold water for 5 minutes. Have your Lab Instructor inspect and assess the burn.
5. The silver nitrate ($AgNO_3$) solution is made up in 4M nitric acid (HNO_3). This solution will cause *both* acid burns and brown or black stains if it contacts your skin. Handle this solution ***only*** while wearing gloves.

Disposal

All aqueous chemicals may be disposed of in the sink and washed down with excess cold water.

Filter papers must be disposed of in the regular trash.

Product crystals and any excess solid reagent should be placed in the "Solid Chemical Waste" carboy.

Background

The the first five experiments in CHEM 103 have introduced many concepts central to working in a laboratory. These include:

- solution preparation and concentration units
- synthesis of compounds both from elements and from other compounds
- mole calculations in synthesis
- limiting reactant calculations and percent yield
- isolation of a product by vacuum filtration
- preparation of standard solutions
- construction and use of a Beer's Law plot

- determination of the concentration of a solution using a Beer's Law plot
- careful acquisition of experimental data that is used to solve a problem

This final experiment of the semester runs over two lab periods. In the first, you will follow a procedure to synthsize and isolate an unknown compound. This material is genuinely new, was prepared for the first time (as far as we know) in the development of this experiment, and has not been reported in the chemical literature. In the second lab period, you will do as synthetic chemists have done for over 200 years, and attempt to characterize the compound you have made so that you can report its composition, and perhaps propose a structure for the product.

Pre-Lab Assignment

1. A chemist prepares a transition metal compound from cobalt(III) chloride and ammonia. The product is sent out for elemental analysis, and the composition is reported back as:

   %Co = 25.2 %C = 0 %H = 5.2 N = 24.0 %Cl = 45.6

   Calculate the empirical formula of the compoound.

2. Do all the calculations necessary to prepare your solutions for steps 1 & 2 of part B of the experiment (page 4).

Preparing Yourself for this Experiment

This experiment involves the synthesis of a chemical compound. You have performed two inorganic syntheses earlier in the semester, so you are expected to be able to measure out and combine chemicals quickly and efficiently. You should be able to carry out the preparation and isolation of the product in under 90 minutes, but this will require that you come to lab fully prepared to do chemistry. Study the sections below and review the ChemPages describing the techniques you will use in performing the synthesis. **Write a summary of the procedure in your lab notebook *before* you come to laboratory**. Your lab notebook should be set up with clear information on what you need, how much of each reagent is required, what you are going to do, and the order in which steps will be performed.

Read the *Techniques* and *Experimental* sections carefully and determine exactly what you will be required to do on the first and second week. Prepare for the first week's work before coming to lab to do the synthesis, and then re-visit the lab outline and prepare yourself for the second week when you will analyze your product.

Techniques in this Experiment

On the Web	In the Manual	In the Textbook
Review the following Laboratory Resource Pages • Balance • Graduated Cylinder • Filtration, Vacuum • Hot plate/Magnetic stirrer	Study the following sections concerning • Gravimetric analysis • Rinsing a solid in a Büchner funnel • Types of observations • Beer's Law • Ocean Optics™ diode array spectrophotometer	Treichel Section 3.6 p101 - 105

Gravimetric analysis

As the name suggests, *gravimetric analysis* involves very accurate weighing of a chemical as a means of working out something about that chemical. A common type of gravimetric analysis involves precipitation of a single component (usually an ion) from a known amount of a compound or mixture. This is accomplished by reacting a solution of the test compound with a solution containing an ion that forms an insoluble salt with the component you wish to test for. In this experiment, chloride ions in the dissolved product will be precipitated as the insoluble salt silver chloride (AgCl) by adding excess silver nitrate to the solution.

Having precipitated the chloride as silver chloride, the AgCl can be filtered off and dried, then weighed accurately. Since there is one mole of chloride ions in one mole of AgCl, converting the mass of AgCl to moles gives the moles of chloride directly. Multiplying the mass of a mole of chloride ions (35.4527 g/mol) by this number of moles gives the mass of chloride ions in the original sample. From this information, you can determine the percent by mass of chloride in your compound.

Although we are analyzing for chloride in this experiment, the general technique is applicable to many systems and analytes. The analytical chemistry courses CHEM 327 and 329 also teach gravimetric analysis, but in more depth and with greater attention to experimental precision and accuracy.

Rinsing a solid in a Büchner funnel

See *Synthesis of an Alum*

Types of observations

See *Reaction Types and Chemical Logic*

Ocean Optics™ diode array spectrophotometer

See *Copper Ammine Compounds*

Experimental

Theory

The compound you will make is formed by the reaction of an organic amine with hydrochloric acid and copper(II) chloride. The product is somewhat soluble in water, so it is important that you keep the concentration high by keeping the volume of solution small. For this reason, you will add solid $CuCl_2 \cdot 2H_2O$ directly to the other reagents, rather than dissolving it in water first as is typically done when reacting two water soluble species. The product is air stable, and will be allowed to dry in your locker between lab periods.

The analysis of your product will rely on determining the percent by mass of each element in the product. You will generate a Beer's Law plot for Cu^{2+} ions in water and use it to find the %mass of Cu. You will determine the %mass of Cl^- gravimetrically as described above.

The most common commercially available form of elemental analysis is CHN micro-analysis. This analysis requires only 2-3 mg of material and reports the percent by mass of carbon, hydrogen and nitrogen in the sample. Many chemical companies and universities have their own CHN facility, and for those that don't there are several companies in the U.S. that offer the service with overnight turnaround for a reasonable cost. We have sent samples of the product you will make out for CHN analysis, and your Lab Instructor will give you those data once you have determined the % Cu and Cl. In addition to Cu, Cl, C, H and N, there is also some oxygen in your product. Note that there may be some experimental error in both your own data and the CHN numbers from the commercial lab. For both these reasons, you should not be concerned if your %mass data do not add exactly to 100%.

Once you have all the %mass data, you will be able to do a calculation to find an empirical formula for your product. You should review empirical formula calculations before coming to the second week of this lab.

Data Collection (Work with a partner)

A Preparing the Compound

Accurately weigh approx. 2 grams of the amine on the analytical balance.

UNDER YOUR STUDENT HOOD. Add the amine to a 100 mL beaker containing 10 mL of 6M HCl. Warm and stir (magnetic) the solution until all the amine has dissolved. This may take several minutes. Note that the hydrochloric acid may give off fumes of HCl gas as it is warmed. It is not necessary to warm the solution very much to get the amine to dissolve, so avoid overheating the solution, as the fuming will increase with temperature.

While the amine is dissolving, accurately weigh approx. 2 grams of $CuCl_2 \cdot 2H_2O$ on the analytical balance. Add this to the stirred amine/HCl solution all at once. Turn off the heat, and continue stirring the solution for 5 minutes, then cool in an ice bath for 10 minutes.

Weigh a filter paper, then collect the product by vacuum filtration. Wash the product twice with 5 mL portions of chilled 6M HCl and twice with 5 mL portions of ethanol. Draw air through the solid to dry it for another 10 minutes, then transfer the product and the filter paper to a weighed watch-glass and store this in your locker to dry until the next lab period.

B Preparing a Beer's Law plot for Cu^{2+}

Review the "Determination of Alcohol Content in Wine" experiment before proceeding. The data analysis and use of the spectrophotometer in the wine laboratory is related to this procedure. Review the "Ocean Optics™ diode array spectrophotometer" technique before using this instrument.

1. Your group should prepare a 0.08 M stock solution of $[Cu(H_2O)_6]^{2+}$ by dissolving copper sulfate pentahydrate in a small amount of dilute sulfuric acid (0.5 M), transferring the solution to a 100 mL volumetric flask, and diluting to the mark with dilute sulfuric acid.
2. Prepare a set of solutions diluted from the stock solution using your graduated pipet and a 10 mL volumetric flask. Your group should prepare *at least* four solutions in the range of 0.010-0.080 M (Divide this work among your group members!). *Note: Each group may have no more than one 10.00 mL volumetric flask per group member.*
3. Measure the absorbance of your solutions at 810 nm. Instructions for using the Ocean Optics™ spectrometers will be distributed in lab. Remember that all solutions are made up in 0.50 M acid. Clean up any spills immediately, expecially those spills that occur in or on the instrument. Pay attention to your Lab Instructor's directions on cleaning up spills.
4. Make a plot of ansorbance (y axis) vs $[Cu^{2+}]$ (x axis) and draw a line of best fit through the points. You may use graph paper or a computer graphing program. This step can be done on your own time outside of the lab, as long as it is completed before the second week of the Project Lab. If one or more of your points do not lie on or very close to the line of best fit you may need to repeat these measurements during the second week in lab.

Do not lose this data! You will need this graph again in CHEM 104

C Recording Experimental Yield

(The following lab day) Weigh the watch-glass, filter paper and product together. Subtract out the weights of the watch-glass and filter paper to find your experimental yield. Consult your instructor immediately if your yield is less than 0.7 gram.

D Finding the Copper Content of the Product

Accurately weigh 0.20g of product (analytical balance). Dissolve this in 0.5M sulfuric acid and make up the volume to the mark in a 25 mL volumetric flask. **Note: use good volumetric technique!** Obtain a *clean* 1cm cuvet and record the absorbance of the solution at 820 nm. Use your Beer's Law plot to find the $[Cu^{2+}]$ in the solution. The copper concentration can be used to calculate the %mass of Cu in your product. The Report Cover Sheet will help you with this calculation.

E Gravimetric Analysis for Chloride

Accurately weigh 0.50g of product (analytical balance) in your smallest beaker. To this, add 5 mL deionized water and swirl (or stir if necessary) to dissolve the product completely.

Add 5 mL 1M $AgNO_3$ in 4M HNO_3 and stir.

Weigh a filter paper, then collect the AgCl precipitate by vacuum filtration. Wash the AgCl twice with 5 mL portions of 4M HNO_3 and twice with 5 mL portions of ethanol. Draw air through the solid to dry it, then transfer the product and the filter paper to a weighed watch-glass.

It is important that the AgCl be as dry as possible. If a drying oven is available in your lab, place your watchglass in it (be sure you know where it is so that you can retrieve the correct one later) at 60°C and dry the product for 20 minutes. If no drying oven is available, dry the AgCl in the Büchner funnel for 20 minutes, then leave it *filter paper side up* on the watchglass for 10 minutes before taking a final weight measurement.

Determine the mass of AgCl precipitated, and convert this to moles of AgCl, and then to moles of chloride. Convert moles of chloride to grams, and find the %mass of chloride in your sample. The Report Cover Sheet will help you with this calculation.

Working with your Data

Record all your data in your lab notebook and on the Report Cover Sheet as you generate the numbers. Calculate the %masses of copper and chlorine in your compound, then show this information to your Lab Instructor. S/he will check your data so far and, if it is reasonable, give you the percent C, H and N as determined by a commercial laboratory *and* the formula of the amine.

ü Check Point

Show your %Cu and %Cl data to your instructor before proceeding.

Use this data together with your experimental data to find the empirical formula for your compound. (Note: don't forget that there is some oxygen in there too, and that the formula you propose must be a charge neutral compound.)

Show your empirical formula to your Lab Instructor, who will initial your Report Cover Sheet. This much must be done before you leave at the end of the lab period. The remaining calculations may be completed on your own time. Your Lab Instructor will inform you of the deadline for submission of your completed report.

The empirical formula for this compound is the same as the molecular formula. Having found the empirical/molecular formula, identify the limiting reagent in the original synthesis, calculate a theoretical yield based on the actual mass of limiting reagent you used, and determine your percent yield in the synthesis.

A Group Report

All data and observations should be organized in *each person's* laboratory notebook. You are required to submit the duplicate page copies of each group member's laboratory notebook for grading.

A Report Cover Page is included at the end of this outline. This page provides spaces for you to organize your data in a uniform format which helps your Lab Instructor to grade all reports fairly and accurately. The cover page is not a replacement for your laboratory notebook. **All data reported on the cover page must first be recorded in your notebook.**

For the Project Lab only, the cover page provides some help with the calculations you need to perform.

You also must complete and attach one Group Report Score Sheet **for each member of your group.**

We hope that you enjoyed CHEM 103 labs, and look forward to seeing you next semester in CHEM 104. Good luck on the final!

The remainder of the points awarded for this lab exercise will be awarded for the following items. Attach the duplicate copies of all pages of your notebook used in this experiment so that your Lab Instructor can assess your work in these areas.

(2 points) **Pre-Lab Preparation.** The following items should be written into your notebook before you arrive at the laboratory: a *short* but detailed outline of the procedure to be followed in the laboratory each day, anticipated space for you to record data.

(2 points) **Notebook Skills.** The duplicate copies of each member's notebook pages must be submitted as part of the report. The notebook will be assessed on how well it meets the *Rules for Keeping a Laboratory Notebook* in the introduction to this manual.

(5 points) **Performance Evaluation.** There is much to do in this experiment, and how competently you perform the tasks will be very clear to your instructor. Participation in the data collection and skill level in previously practiced techniques will be actively evaluated.

Project Lab: Lab Report Cover Page

Complete one report per group. Attach copies of all pertinent lab notebook pages to these sheets.

Fill in your section number and names.

Your Names_____ & _____ Section No._____ Report Score_____

Organize your observations for your Lab Instructor

Observations about chemicals

Chemical	Observations

Organize your data for your Lab Instructor

Mass of amine used in synthesis_____g = _____ mol amine (Mol. mass = 135.17 g/mol)

Volume of 6M HCl used in synthesis_____mL = _____ mol HCl

Mass of $CuCl_2 \cdot 2H_2O$ used in synthesis_____g = _____ mol $CuCl_2 \cdot 2H_2O$

Experimental yield of product_____g

Mass of product dissolved for Cu determination_____g

Mass of product dissolved for Cl determination_____g

Mass of AgCl recovered in gravimetric analysis_____g

Continued on reverse……..

Calculate the %copper in the product

Measured absorbance of Cu solution _____ABS units

Concentration of Cu solution from Beer's Law Plot, $[Cu^{2+}]$ = _____mol/L

$[Cu^{2+}]$ x volume of solution: _____ mol/L x 0.01000 L = _____mol Cu

mol. Cu x mol. mass Cu: _____mol x 63.546 g/mol = _____ g Cu

$\frac{\text{\_\_\_\_\_g Cu\_\_\_\_\_}}{\text{mass of product used}} \times 100 : \frac{\text{\_\_\_\_\_g}}{\text{g}} \times 100 =$ _____**%copper**

Calculate the %chlorine in the product

$\frac{\text{Mass of AgCl recovered}}{\text{mol. mass AgCl:}} : \frac{\text{\_\_\_\_\_g}}{143.3209\text{g/mol}} =$ _____mol. AgCl = _____mol. Cl

mol. Cl x mol. mass Cl: _____ mol x 35.4527 g/mol = _____g Cl

$\frac{\text{\_\_\_\_\_g Cl\_\_\_\_\_}}{\text{mass of product used}} \times 100 : \frac{\text{\_\_\_\_\_g}}{\text{g}} \times 100 =$ _____**%chlorine**

Enter the CHN and formula information supplied by your Lab Instructor here:

%C_____ %H_____ %N_____ formula of amine _____

Calculate the empirical formula of the compound you synthesized.

The remaining calculations may be performed outside of lab. Show your calculations in the space below and overleaf, and/or on additional sheets of paper, then insert the numeric answers on the lines that follow:

Based on the empirical formula, calculate the **molar mass of your product**._____ g/mol (*Reminder: the empirical formula is the* same *as the molecular formula in this case.*)

Write a balanced chemical equation for the reaction that you carried out to make the product.

Using the stoichiometry of the product (i.e. how many moles of amine, copper and chlorine, respectively are in it) and the amounts of reagent you used in your synthesis, determine which reagent was limiting.

Limiting reagent was _____

Calculate the theoretical yield based on the amount of limiting reagent used.

Theoretical yield = _____g

Calculate your percentage yield. **Percent yield** = _____%