

Preparing Tetraamminecopper(II) Sulfate Monohydrate

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Purpose of the Experiment

Prepare tetraamminecopper(II) sulfate monohydrate by reacting copper(II) sulfate pentahydrate and ammonia in aqueous solution. Isolate the product and determine the percent yield.

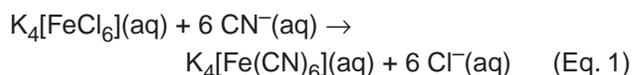
Background Information

Coordination compounds are an important class of inorganic substances that contain a metal ion to which other ions or molecules are attached by means of coordinate covalent bonds. The ions or molecules that are bonded to the metal ion are called **ligands**. The central metal ion and attached ligands form a **complex ion**, or **complex**. The simplest complexes are the hydrated cations of the transition metals. For example, in many common salts, such as $\text{Co}(\text{NO}_3)_2 \cdot 6 \text{H}_2\text{O}$ and $\text{FeSO}_4 \cdot 7 \text{H}_2\text{O}$, the metal ion is bonded to six water molecules to form the hexaaqua complex ion, $[\text{M}(\text{H}_2\text{O})_6]^{2+}$, where M represents the metal cation. The hexaaqua complex is the dominant species present in aqueous solutions of these salts. In the case of copper(II) sulfate pentahydrate, $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$, the complex has four water molecules bonded to the metal.

Many different molecules and ions can function as ligands in coordination compounds. Complex formation results from ion-ion or ion-dipole interactions between a positively charged cation and either anions or the negative part of polar molecules. The primary requirement for ligands is that they have at least one lone electron pair capable of interacting with empty metal

ion orbitals. Commonly encountered ligands include halide ions (F^- , Cl^- , Br^- , and I^-), cyanide ion (CN^-), carbon monoxide (CO), water (H_2O), and ammonia (NH_3). The coordinate covalent bonds in a complex are generally relatively weak. As a result, one or more of the ligands may be replaced in simple single replacement reactions.

Consider the reaction described by Equation 1.



Potassium hexachloroferrate(II), $\text{K}_4[\text{FeCl}_6]$, contains the hexachloroferrate(II) complex ion, $[\text{FeCl}_6]^{4-}$. This complex consists of an iron(II) ion (Fe^{2+}) with six Cl^- ions bonded to it in an octahedral arrangement. Note that the overall charge on the complex ion is the sum of the charges on the central metal ion and the ligands. Because the $[\text{FeCl}_6]^{4-}$ complex has a 4- charge, four potassium ions (K^+) are necessary for charge balance in the neutral compound. Thus, the formula of potassium hexachloroferrate(II) is $\text{K}_4[\text{FeCl}_6]$.

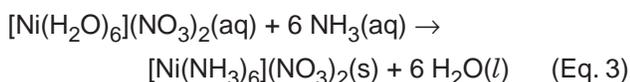
To perform the reaction shown in Equation 1, we add an aqueous solution of CN^- ion to a solution of $\text{K}_4[\text{FeCl}_6]$. The CN^- ions replace the Cl^- ions originally

bound to the iron(II) ion, producing the hexacyanoferrate(II) ion, $[\text{Fe}(\text{CN})_6]^{4-}$ in solution. We can then crystallize the complex as the potassium hexacyanoferrate(II) salt, $\text{K}_4[\text{Fe}(\text{CN})_6]$.

As with any chemical synthesis, we are interested in the mass of product we can prepare from such a reaction. This mass depends on several factors, the most important of which are the masses of the reactants involved and the reaction stoichiometry. For many reactions, we limit the amount of one reactant and add the others in excess. Such a reaction ceases when the **limiting reactant** or **reagent** has completely reacted. We calculate the maximum possible yield of the reaction, the **theoretical yield**, by assuming that the limiting reagent reacts completely to form the product. In practice, the mass of product we actually obtain, the **actual yield**, is generally somewhat smaller than the theoretical yield. This usually happens because it is difficult to optimize all the reaction conditions, such as temperature and solution concentrations. Also, we inevitably lose some product during the physical manipulations involved in isolating it from the reaction mixture. The **percent yield** of the reaction, which we calculate from the actual and theoretical yields as shown in Equation 2, is an expression of the efficiencies of the reaction, the procedure, and our technique. The percent yield is generally less than 100%. Due to the law of conservation of mass, the yield cannot exceed 100%.

$$\text{percent yield} = \left(\frac{\text{actual yield, g}}{\text{theoretical yield, g}} \right) (100\%) \quad (\text{Eq. 2})$$

Consider the following example, in which we synthesize hexaamminenickel(II) nitrate by reacting hexa-aquanickel(II) nitrate and NH_3 in aqueous solution, as shown in Equation 3.



Suppose we dissolved in water 4.235 g of $[\text{Ni}(\text{H}_2\text{O})_6](\text{NO}_3)_2$ and added 20.0 mL of concentrated NH_3 solution (9.0M). We then isolated 3.886 g of $[\text{Ni}(\text{NH}_3)_6](\text{NO}_3)_2$ from the reaction mixture. To determine the percent yield of the reaction, we first use Equation 4 to calculate the number of moles of each reagent added.

number of moles of compound, mol =

$$\left(\frac{\text{mass of compound, g}}{\text{molar mass of compound, g}} \right) \left(\frac{1 \text{ mol of compound}}{1 \text{ mol of compound}} \right) \quad (\text{Eq. 4})$$

number of moles of $[\text{Ni}(\text{H}_2\text{O})_6](\text{NO}_3)_2$ added, mol =

$$\left(\frac{4.235 \text{ g}}{[\text{Ni}(\text{H}_2\text{O})_6](\text{NO}_3)_2} \right) \left(\frac{1 \text{ mol } [\text{Ni}(\text{H}_2\text{O})_6](\text{NO}_3)_2}{290.79 \text{ g } [\text{Ni}(\text{H}_2\text{O})_6](\text{NO}_3)_2} \right) = 1.456 \times 10^{-2} \text{ mol } [\text{Ni}(\text{H}_2\text{O})_6](\text{NO}_3)_2$$

and

number of moles of NH_3 added, mol =

$$\left(\frac{20.0 \text{ mL}}{\text{NH}_3 \text{ solution}} \right) \left(\frac{1 \text{ L}}{1000 \text{ mL}} \right) \left(\frac{9.0 \text{ mol NH}_3}{1 \text{ L NH}_3 \text{ solution}} \right) = 0.18 \text{ mol NH}_3$$

Then we identify the limiting reagent. According to Equation 3, six moles of NH_3 react with each mole of $[\text{Ni}(\text{H}_2\text{O})_6](\text{NO}_3)_2$. Therefore, because we added 1.456×10^{-2} mol of $[\text{Ni}(\text{H}_2\text{O})_6](\text{NO}_3)_2$, we need six times as many moles of NH_3 , or 8.736×10^{-2} mol, for complete reaction of the $[\text{Ni}(\text{H}_2\text{O})_6](\text{NO}_3)_2$. We actually added 0.18 mol of NH_3 , which is more than twice the amount needed to completely react with the $[\text{Ni}(\text{H}_2\text{O})_6](\text{NO}_3)_2$ present. Because NH_3 was present in excess, $[\text{Ni}(\text{H}_2\text{O})_6](\text{NO}_3)_2$ is the limiting reagent.

We calculate the theoretical yield, in moles, of product from the number of moles of limiting reagent and the stoichiometry of Equation 5.

theoretical yield, mol =

$$\left(\frac{\text{number of moles of limiting reagent, mol}}{\text{stoichiometric coefficient of limiting reagent}} \right) \left(\frac{\text{stoichiometric coefficient of product}}{\text{stoichiometric coefficient of limiting reagent}} \right) \quad (\text{Eq. 5})$$

theoretical yield of $[\text{Ni}(\text{NH}_3)_6](\text{NO}_3)_2$, mol =

$$\left(\frac{1.456 \times 10^{-2} \text{ mol}}{[\text{Ni}(\text{H}_2\text{O})_6](\text{NO}_3)_2} \right) \left(\frac{1 [\text{Ni}(\text{NH}_3)_6](\text{NO}_3)_2}{1 [\text{Ni}(\text{H}_2\text{O})_6](\text{NO}_3)_2} \right) = 1.456 \times 10^{-2} \text{ mol } [\text{Ni}(\text{NH}_3)_6](\text{NO}_3)_2$$

We calculate theoretical yield, in grams, from the theoretical yield in moles and the molar mass of the product, using Equation 6.

theoretical yield, g =

$$\left(\frac{\text{theoretical yield, mol}}{\text{yield, mol}} \right) \left(\frac{\text{molar mass of product, g}}{1 \text{ mol of product}} \right) \quad (\text{Eq. 6})$$

theoretical yield of $[\text{Ni}(\text{NH}_3)_6](\text{NO}_3)_2$, g =

$$\left(\frac{1.456 \times 10^{-2} \text{ mol}}{[\text{Ni}(\text{NH}_3)_6](\text{NO}_3)_2} \right) \left(\frac{284.88 \text{ g } [\text{Ni}(\text{NH}_3)_6](\text{NO}_3)_2}{1 \text{ mol } [\text{Ni}(\text{NH}_3)_6](\text{NO}_3)_2} \right) = 4.148 \text{ g } [\text{Ni}(\text{NH}_3)_6](\text{NO}_3)_2$$

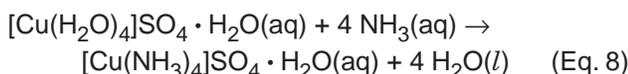
Finally, we calculate the percent yield from the actual and theoretical yields, using Equation 2.

$$\text{percent yield} = \left(\frac{\text{actual yield, g}}{\text{theoretical yield, g}} \right) (100\%) \quad (\text{Eq. 2})$$

percent yield of $[\text{Ni}(\text{NH}_3)_6](\text{NO}_3)_2$, g =

$$\left(\frac{3.886 \text{ g } [\text{Ni}(\text{NH}_3)_6](\text{NO}_3)_2}{4.148 \text{ g } [\text{Ni}(\text{NH}_3)_6](\text{NO}_3)_2} \right) (100\%) = 93.68\%$$

In this experiment, you will prepare tetraamminecopper(II) sulfate monohydrate, $[\text{Cu}(\text{NH}_3)_4]\text{SO}_4 \cdot \text{H}_2\text{O}$, from a reaction of $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$ and NH_3 in aqueous solution. Although we usually write the formula of the reactant compound as $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$, in the crystalline state this compound is composed of tetraaquacopper(II) complex ions, $[\text{Cu}(\text{H}_2\text{O})_4]^{2+}$. Therefore, we might better represent the compound as $[\text{Cu}(\text{H}_2\text{O})_4]\text{SO}_4 \cdot \text{H}_2\text{O}$. The fifth water molecule is not part of the complex ion, but is hydrogen bonded to the sulfate ion (SO_4^{2-}) in the solid. The chemical reaction described above is shown in Equation 8.



This is a **single replacement reaction** in which NH_3 molecules replace the water molecules originally bonded to the copper(II) ion.

The strategy you will employ in this synthesis is to add a large excess of NH_3 solution to an aqueous solution of $[\text{Cu}(\text{H}_2\text{O})_4]^{2+}$ ion in order to achieve a complete replacement of H_2O by NH_3 . At this point, you will have the product in solution, where it exists as discrete tetraamminecopper(II) ions, $[\text{Cu}(\text{NH}_3)_4]^{2+}$, and SO_4^{2-} ions. To isolate the product as a solid, you must crystallize it. To do this, you will evaporate a portion of the water, add some ethanol, and cool the solution in an ice bath. Each of these steps reduces the solubility of the product and is therefore important in maximizing your yield. Because ethanol is less polar than water, ethanol is a poor solvent for ionic compounds. Addition of ethanol to the solution reduces the solubility of $[\text{Cu}(\text{NH}_3)_4]\text{SO}_4 \cdot \text{H}_2\text{O}$ in the mixture, causing the product to crystallize. Very little, if any, solid $[\text{Cu}(\text{NH}_3)_4]\text{SO}_4 \cdot \text{H}_2\text{O}$ will form if you do not add ethanol. If you fail to reduce the water volume or to cool the solution sufficiently, your yield will also be reduced, because some of the $[\text{Cu}(\text{NH}_3)_4]\text{SO}_4 \cdot \text{H}_2\text{O}$ will remain dissolved in solution.

You can also help maximize your yield by using proper experimental technique. You must be particularly careful to prevent loss of product during certain

steps in the synthesis. First, when evaporating the water, you must heat the $\text{CuSO}_4\text{--NH}_3$ solution gently. Some of the $[\text{Cu}(\text{NH}_3)_4]\text{SO}_4 \cdot \text{H}_2\text{O}$ may decompose if you heat the reaction mixture too vigorously. If some decomposition does occur, you will see a brown film on the bottom of your beaker and a blackish brown tinge in the solid product.

Second, you must be careful when you filter the crystallized product from the supernatant liquid. The filtration process involves three steps: decanting the supernatant liquid, transferring the solid product, and washing the solid product. **Decantation** is the process of pouring the supernatant liquid into the funnel without disturbing the solid at the bottom of the beaker. The filtration takes longer if you do not transfer most of the liquid to the funnel **before** you begin to transfer the solid. Once this is done, you can pour the wet solid into the funnel and use a rubber policeman attached to a glass stirring rod to scrape the remaining solid out of the beaker.

Clearly, you want to transfer **all** of the product from the beaker into the funnel. To assist in getting the last remaining product out of the beaker, you will add a small amount of ice-cold NH_3 –ethanol solution to the beaker, swirl, and immediately transfer the mixture to the funnel. Then you will wash the solid with the remainder of this ice-cold solution as well as with a portion of ice-cold ethanol. It is important that the washing solutions be very cold and free of water so you do not redissolve any of the product.

Finally, you will dry and weigh your product. You will then calculate the theoretical and percent yields for your synthesis.

Procedure

Chemical Alert

concentrated ammonia—toxic and corrosive
copper(II) sulfate pentahydrate—toxic and irritant
ethanol—toxic and flammable

Caution: Wear departmentally approved eye protection while doing this experiment.

I. Preparing and Mixing the Reactants

Note: If weighing boats are not available in your laboratory, your laboratory instructor will demonstrate and describe a satisfactory method for

weighing and transferring the solids used in this experiment.

Unless your laboratory instructor tells you otherwise, record all masses to the nearest milligram (0.001 g).

1. Weigh a clean, dry weighing boat. Record the mass of the weighing boat on your Data Sheet.

Weigh about 6.0 g of $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$ into the weighing boat. Record the mass of the weighing boat and $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$ on your Data Sheet.

Transfer the solid into a clean 250-mL beaker.

Caution: Concentrated NH_3 solution is toxic, corrosive, and irritating. Prevent contact with your eyes, skin, and clothing. Permanent fogging of soft contact lenses may result from NH_3 vapors. Avoid inhaling vapors and ingesting the solution.

If you spill any NH_3 solution on yourself, immediately rinse with a large amount of running water. If you spill any solution on the laboratory bench, add water and wipe up immediately with a damp paper towel. Immediately notify your laboratory instructor of any NH_3 spills.

Carry out Steps 2 and 3 under a fume hood.

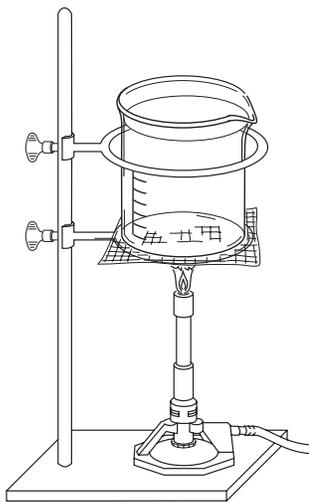


Figure 1 Heating a beaker

2. Add 20 mL of distilled or deionized water to the solid in the beaker. Place the beaker through a ring attached to a ring stand, as shown in Figure 1. Rest the beaker on a ceramic-centered wire gauze on a second ring attached to the ring stand **making sure that the beaker is at least partially under a fume hood.**

Gently heat the beaker and its contents with the flame from a Bunsen burner until the solid completely dissolves. **Do not** heat the solution to boiling.

Note: As you add the NH_3 solution to the copper(II) sulfate solution in Step 3, you may observe the formation of a thick, light blue precipitate of copper hydroxide, $\text{Cu}(\text{OH})_2$. This solid should completely disappear when you add all of the NH_3 solution.

3. In the fume hood, measure 20 mL of concentrated NH_3 solution into a 100-mL graduated cylinder. Record the molarity of the NH_3 solution on your Data Sheet.

After all of the solid in the beaker has dissolved, slowly add the NH_3 solution to the solution in the beaker. Stir the reaction mixture with a glass stirring rod after each 1–2 mL addition of NH_3 solution.

After you have added all of the NH_3 solution, increase the heat and bring the solution to a **very gentle** boil. At this point you should have about 40 mL of a deep purple-violet solution. Continue to boil the reaction mixture until the solution volume has been reduced to approximately 25 mL.

Caution: Ethanol is highly flammable. Be sure that the gas flow to your Bunsen burner is shut off, and that there are no other open flames nearby when you bring the ethanol container to your work area.

Note: Ammonia vapors are very irritating. Be careful not to leave open NH_3 reagent bottles or other containers of NH_3 outside the hood. Your laboratory instructor will tell you whether or not it is necessary for you to stopper the test tube containing the NH_3 –ethanol mixture.

Caution: Do not begin Step 4 until your laboratory instructor informs the whole class to do so. There should be no open flames in the laboratory when everyone is doing Step 4.

4. Fill a 400-mL beaker two-thirds full with ice and water to prepare an ice-water bath. In a labeled, clean, and dry 25 × 250-mm test tube, combine 20 mL of

concentrated NH_3 solution and 20 mL of ethanol. Transfer 25 mL of ethanol to another, labeled, clean, and dry 25×250 -mm test tube. Place these test tubes in the ice-water bath to cool.

5. When the reaction mixture in the beaker (Step 3) has evaporated down to 25 mL, turn off the gas flow and extinguish the flame. Allow the beaker to cool to the point at which you can comfortably touch it with your hand. Carefully remove the beaker from the ring stand and set it on the bench top.

When the beaker has cooled to almost room temperature, add 20 mL of room-temperature ethanol (not the chilled ethanol prepared in Step 4).

6. Fill an 800-mL beaker about two-thirds full with ice and water. Nest the 250-mL beaker containing the reaction mixture in this ice-water bath to cool for at least 15 min.

Note: At this point you should notice a deep blue-violet solid settling to the bottom of the beaker. The appearance of the colorless supernatant liquid above the solid is a good indication that all of the product has crystallized from the solution.

II. Isolating the Product by Vacuum Filtration

Note: Your laboratory instructor will inform you whether you are to use the Büchner funnel with a bored rubber stopper or with a rubber adapter especially designed for this purpose.

If your aspirator does not have a trap attached to it as shown in Figure 2, your laboratory instructor will give you information for assembling the trap.

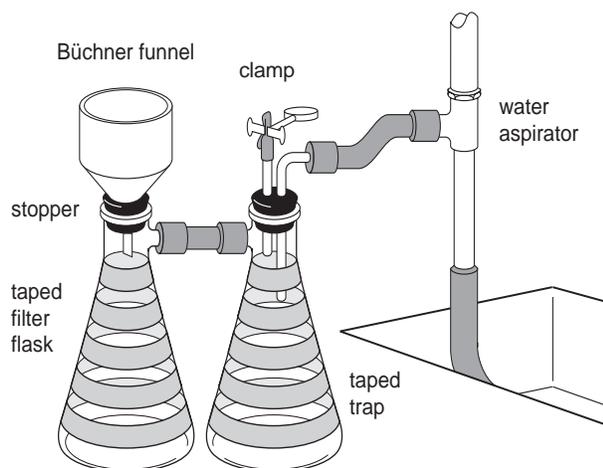


Figure 2 A vacuum filtration apparatus

7. Set up the filtration assembly shown in Figure 2. Either place the Büchner funnel with a rubber stopper in the filter flask, or place a rubber adapter on top of the filter flask, and then place the Büchner funnel on the adapter.

Place a flat circle of filter paper in the funnel. The paper should lie flat in the bottom of the funnel and cover all the holes. To ensure an airtight seal between the filter paper and the funnel, wet the paper with distilled water, and turn on the water aspirator to remove any excess water. You may have to press down on the open top of the Büchner funnel with the palm of your hand in order to make a seal and create a vacuum. Do so carefully, especially if your flask is not clamped and seated on a level surface. Make sure that the paper is damp and flat when you start your filtration.

Note: Be sure to observe the appearance of the filtrate (the liquid in the filter flask) while you are filtering. Remember that the product is blue-violet and the supernatant liquid, a mixture of NH_3 solution and ethanol, is colorless. Consult your laboratory instructor if the filtrate is blue.

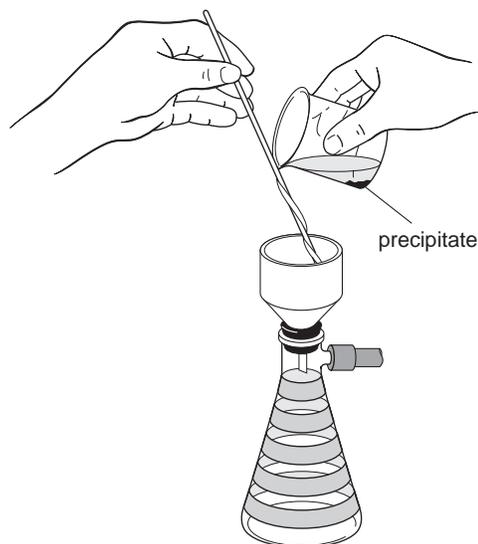


Figure 3 Decanting a supernatant liquid from a beaker to a Büchner funnel

8. Decant as much of the supernatant liquid as possible into the Büchner funnel. To prevent splashing and product loss, use a glass stirring rod to guide the liquid from the beaker onto the filter paper, as shown in Figure 3.

After you have decanted most of the liquid, pour the wet solid onto the filter paper. Use a rubber policeman attached to your glass stirring rod to scrape as much of the solid as possible into the funnel. To

transfer the remaining solid from the beaker, rinse the beaker with up to 5 mL of the ice-cold NH_3 -ethanol solution you prepared in Step 4. Pour the rinses into the Büchner funnel.

Turn on the water aspirator and draw the supernatant liquid from the product through the filter paper. After a few minutes, open the clamp on the top of the trap and turn off the aspirator.

9. Wash the solid with about 5 mL of the ice-cold NH_3 -ethanol mixture. Reclose the clamp, turn on the aspirator, and draw the wash solution through the filter.

Repeat the washing procedure 3 or 4 more times. Then, wash the solid with about 5 mL of the chilled ethanol you prepared in Step 4.

Repeat the ethanol washing 2 or 3 times. After the last washing, dry the solid by leaving the aspirator on to pull air through filter funnel. Test for dryness by carefully turning over the solid with a spatula. The dry solid should powder easily and should not be clumpy. When the product appears dry by this test, turn off the aspirator and remove the Büchner funnel.

10. Transfer the filtrate into the "Discarded Copper Solutions" container.

III. Processing the Product

11. Weigh a clean, dry weighing boat or watch glass to the nearest 0.001 g. Record this mass on Part III of your Data Sheet. Carefully transfer the solid product from the Büchner funnel to the weighing boat. Use a spatula to scrape as much solid as possible from the interior of the funnel and the filter paper.

12. Allow the product to air dry as time permits. Weigh the weighing boat and dry solid to the nearest milligram. Record this mass on your Data Sheet.

13. Transfer your product into the container provided by your laboratory instructor and labeled "Discarded $[\text{Cu}(\text{NH}_3)_4]\text{SO}_4 \cdot \text{H}_2\text{O}$."

Caution: Wash your hands thoroughly with soap or detergent before leaving the laboratory.

Calculations

(Do the following calculations and record the results on your Data Sheet.)

1. Calculate the number of moles of $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$ in the reaction mixture, based on the mass of $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$ you used.
2. Calculate the number of moles of NH_3 in the reaction mixture, based on the volume of NH_3 you used and the molarity of the solution.
3. Use the appropriate calculations to determine the limiting reagent for this synthesis. Write the formula of the limiting reagent on your Data Sheet.
4. Based on the identity of the limiting reagent and the stoichiometry of the reaction, calculate the theoretical yield of $[\text{Cu}(\text{NH}_3)_4]\text{SO}_4 \cdot \text{H}_2\text{O}$ in grams.
5. Use the actual and theoretical yields to calculate the percent yield of your synthesis.

Post-Laboratory Questions

(Use the spaces provided for the answers and additional paper if necessary.)

1. Your percent yield of $[\text{Cu}(\text{NH}_3)_4]\text{SO}_4 \cdot \text{H}_2\text{O}$ is dependent on the quality of your laboratory technique. Give three examples of errors in technique that would lower the percent yield of the synthesis. Briefly explain how each error would lower the percent yield.

(1)

(2)

(3)

2. Two students prepared $[\text{Cu}(\text{NH}_3)_4]\text{SO}_4 \cdot \text{H}_2\text{O}$ using the procedure described in this module. One student used good technique throughout, was careful not to lose product, and obtained a yield of 93.4%. The other student was not nearly as careful, lost noticeable amounts of product during the experiment, yet obtained a yield of 98.6%. How might you explain these results? Do you think that the 98.6% yield is valid? What experimental error might cause a percent yield to be incorrectly high?

3. (1) A coordination compound has the general formula $\text{Pt}(\text{NH}_3)_4\text{Cl}_4$. When this compound dissolves in water, it dissociates into three ions. One of the three ions is an octahedral complex in which the Pt(IV) ion has six ligands bonded to it. What is the formula of the complex ion?

(2) If a solution of this compound is treated with NH_3 , a new compound with the general formula $\text{Pt}(\text{NH}_3)_5\text{Cl}_4$ can be crystallized. When this compound is redissolved in water, it dissociates into four ions. One

of the four ions is an octahedral complex in which the Pt(IV) ion has six ligands bonded to it. What is the formula of this new complex?

(3) In (2), what change has taken place in the original complex to produce the new complex?

Data Sheet

I. Preparing and Mixing the Reactants

mass of weighing boat and $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$, g _____

mass of weighing boat, g _____

mass of $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$, g _____

molarity of ammonia solution, M _____

III. Processing the Product

mass of weighing boat and $[\text{Cu}(\text{NH}_3)_4]\text{SO}_4 \cdot \text{H}_2\text{O}$, g _____

mass of weighing boat, g _____

mass of product, g _____

number of moles $\text{CuSO}_4 \cdot 5 \text{H}_2\text{O}$ added, mol _____

number of moles of NH_3 added, mol _____

formula of the limiting reagent _____

theoretical yield of $[\text{Cu}(\text{NH}_3)_4]\text{SO}_4 \cdot \text{H}_2\text{O}$, g _____

percent yield of $[\text{Cu}(\text{NH}_3)_4]\text{SO}_4 \cdot \text{H}_2\text{O}$, % _____

Pre-Laboratory Assignment

1. Briefly describe the hazards associated with using the following.

(1) concentrated NH_3 solution

(2) ethanol

2. Define the following terms as they pertain to this experiment.

(1) complex ion

(2) ligand

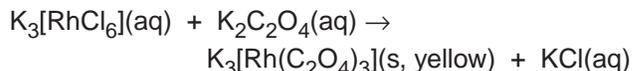
(3) limiting reagent

(4) theoretical yield

(5) percent yield

3. We can prepare the yellow coordination compound potassium tris(oxalato)rhodate(III), $\text{K}_3[\text{Rh}(\text{C}_2\text{O}_4)_3]$, by reacting potassium hexachlororhodate(III), $\text{K}_3[\text{RhCl}_6]$, and potassium oxalate, $\text{K}_2\text{C}_2\text{O}_4$.

(1) Balance the chemical equation for this reaction, shown below.



(2) If we combine 1 mol of $\text{K}_3[\text{RhCl}_6]$ with 1 mol of $\text{K}_2\text{C}_2\text{O}_4$, which compound is the limiting reagent?

(3) How many moles of $\text{K}_3[\text{Rh}(\text{C}_2\text{O}_4)_3]$ could form from the reaction in (2)?

4. A student, using the reaction in 3.(1), combined 1.00 g of $\text{K}_3[\text{RhCl}_6]$ with 25.0 mL of 1.25M $\text{K}_2\text{C}_2\text{O}_4$, and isolated 0.96 g of $\text{K}_3[\text{Rh}(\text{C}_2\text{O}_4)_3]$.

(1) Calculate the number of moles of $\text{K}_3[\text{RhCl}_6]$ the student used.

(2) Calculate the number of moles of $\text{K}_2\text{C}_2\text{O}_4$ the student used.

(3) Determine which reactant is the limiting reagent. Include all appropriate calculations.

(2) Add ethanol to the reaction mixture.

(4) Calculate the theoretical yield of the synthesis, in grams, of $K_3[Rh(C_2O_4)_3]$.

(3) Keep the ethanol vapors away from Bunsen burner flames.

(5) Calculate the percent yield of the synthesis.

(4) Chill the reaction mixture.

5. Several of the procedural steps you will use in this experiment are listed below. Briefly explain the purpose of each step.

(1) Gently heat the reaction mixture.

(5) Wet the filter paper before beginning the filtration.