AP Lab #15 – Synthesis of a Coordination Compound and Its Chemical Analysis

Lab Objectives

In this lab you will:

- → Synthesize a coordination compound.
- → Perform a filtration and crystallization.
- → Understand Lewis acid-base theory.
- → Determine percent yield.

Overview

In this lab, a coordination compound will be synthesized from copper(II) sulfate and ammonia. A coordination compound is a metal compound that is bound to surrounding covalent molecules or ions. In this reaction, water molecules will be removed from copper(II) sulfate pentahydrate, and ammonia will replace them.

Background

Coordination compounds are a highly useful class of molecules. The molecule heme is a coordination compound involving iron. Heme is the oxygen-carrying component of hemoglobin in our blood. Plants use a magnesium coordination compound, chlorophyll, to carry out photosynthesis.

Coordination compounds are composed of a centrally located transition metal surrounded by covalent molecules or ions called ligands. These ligands will share a pair of electrons with the metal center, and in this way it can be said that the metal is a Lewis acid, and the ligands are Lewis bases.

In general, the interactions between the metal and the ligands have energies corresponding to visible regions of light. These complexes are often intensely colored because of those energy levels. Strong interactions tend to yield high energy absorbance. Weak interactions tend to yield low energy absorbance.

In this experiment, copper(II) sulfate pentahydrate (systematically named tetraaquacopper(II) sulfate monohydrate) will react with ammonia to form tetraamminecopper(II) sulfate monohydrate by the reaction:

$$CuSO_4 \cdot (H_2O)_5(aq) + 4NH_3(aq) \longrightarrow [Cu(NH_3)_4]SO_4 \cdot H_2O(aq) + 4H_2O(l)$$

The color of the initial solution and product are dramatic, and their difference clearly indicates a reaction has occurred.

The driving force for this reaction is ligand field strength. This describes the strength of the interactions between the metal atom and its ligands. A ligand with weak field strength will separate from a metal if a ligand with strong field strength is present to take its place. The more strongly a ligand interacts with the metal, the more stable the resulting compound is.

Student Guide – Kit #9591403

Sample Calculation

A student attempted to synthesize the coordination compound $[Cr(H_2O)_5CN]$. This reaction describes its formation:

$$[Co(NH_3)_5]CN + Cr(H_2O)_6 + 5H_2O \longrightarrow Co(H_2O)_6 + [Cr(H_2O)_5]CN + 5NH_3$$

The following amounts of reactants were used:

- 3.91 g $[Co(NH_3)_5]CN$
- 5.44 g $Cr(H_2O)_6$
- 4.22 g H_2O

Using this data, determine the following:

1. What is the limiting reagent in this experiment?

$$3.91 \text{ g } [Co(NH_3)_5]CN \times \frac{1 \text{ mol } [Co(NH_3)_5]CN}{170.10 \text{ g } [Co(NH_3)_5]CN} = 0.023 \text{ mol } [Co(NH_3)_5]CN$$

$$5.44 \text{ g } Cr(H_2O)_6 \times \frac{1 \text{ mol } Cr(H_2O)_6}{160.09 \text{ g } Cr(H_2O)_6} = 0.034 \text{ mol } Cr(H_2O)_6$$

$$4.22 \text{ g } H_2O \times \frac{1 \text{ mol } H_2O}{18.02 \text{ g } H_2O} = 0.234 \text{ mol } H_2O$$

$$\frac{0.234 \text{ mol } H_2O}{5} = 0.047 \text{ mol}$$

Note: This accounts for the mole ratio of 1:1:5.

$[Co(NH_3)_5]CN$ is the limiting reagent.

2. Using the limiting reagent, what is the theoretical yield of $[Cr(H_2O)_5]CN$, in grams, of this experiment?

$$0.023 \text{ mol } [Co(NH_3)_5]CN \times \frac{1 \text{ mol } [Cr(H_2O)_5]CN}{1 \text{ mol } [Co(NH_3)_5]CN} = 0.023 \text{ mol } [Cr(H_2O)_5]CN$$

$$0.023 \text{ mol } [Cr(H_2O)_5]CN \times \frac{168.09 \text{ g } [Cr(H_2O)_5]CN}{1 \text{ mol } [Cr(H_2O)_5]CN} = 3.87 \text{ g } [Cr(H_2O)_5]CN$$

Sample Calculation (continued)

3. The student found the actual yield for $[Cr(H_2O)_5]CN$ was 2.92 g. What was the percent yield of this experiment?

$$\frac{2.92 \text{ g } [Cr(H_2O)_5]CN}{3.87 \text{ g } [Cr(H_2O)_5]CN} \times 100 = 75.5\%$$

4. Based on the student's yield of $[Cr(H_2O)_5]CN$, what mass of $Co(H_2O)_6$ should have been produced?

$$2.92 \text{ g } \cancel{[Cr(H_2O)_5]CN} \times \frac{1 \text{ mol } [Cr(H_2O)_5]CN}{168.09 \text{ g } \cancel{[Cr(H_2O)_5]CN}} = 0.017 \text{ mol } [Cr(H_2O)_5]CN$$

$$0.017 \text{ } \cancel{\text{mol } [Cr(H_2O)_5]CN} \times \frac{1 \text{ mol } Co(H_2O)_6}{1 \text{ } \cancel{\text{mol } [Cr(H_2O)_5]CN}} = 0.017 \text{ mol } Co(H_2O)_6$$

$$0.017 \text{ } \cancel{\text{mol } Co(H_2O)_6} \times \frac{167.02 \text{ g } Co(H_2O)_6}{1 \text{ } \cancel{\text{mol } Co(H_2O)_6}} = 2.90 \text{ g } Co(H_2O)_6$$

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Pre-Lab Questions

Synthesis of a Coordination Compound

Answering the following questions will help prepare for the concepts covered in this lab.

1. Determine the mass of $CuSO_4 \cdot 5H_2O$ required to produce 2.0 grams of $[Cu(NH_3)_4]SO_4 \cdot H_2O$, assuming an 85% yield.

2. Determine the amount of 2.3 M ammonia required to react with the amount of $CuSO_4 \cdot 5H_2O$ in Question 1.

3. If 3.50 g of $[Cu(NH_3)_4]SO_4 \cdot H_2O$ is prepared from 4.25 g of $CuSO_4 \cdot 5H_2O$, what is the percent yield?

4. Given that this reaction is spontaneous (requires no heat to react), which ligand forms a more stable coordination compound with copper, H_2O or NH_3? Explain.

AP Lab #15 – Synthesis of a Coordination Compound

Materials

Chemicals

→ Ammonia, NH_4OH, 2.3 M, 50 mL

→ Copper(II) sulfate pentahydrate, $CuSO_4 \cdot 5H_2O$, 3–5 g

→ Ethanol, denatured, 50 mL

→ DI water, 15 mL

Lab Supplies and Equipment

→ Filter paper

→ Beaker, 100-mL

→ Bunsen burner or hot plate

→ Filter flask, 250-mL, only if using Buchner funnel

→ Funnel, short-stem filter or Buchner

→ Graduated cylinder, 25-mL

→ Graduated cylinder, 50-mL

→ Plastic or rubber tubing, only if using Buchner funnel

→ Ring stand with ring

→ Stir rod with rubber or plastic policeman

→ Tongs

Safety

→ Read all instructions thoroughly before you begin.

→ Wear safety goggles at all times during this experiment.

→ Ethanol is extremely flammable, and toxic by inhalation and ingestion. Keep away from open flames. Avoid breathing vapors.

→ Concentrated ammonia solution is toxic by ingestion and inhalation, and is irritating to the skin. Avoid breathing vapors.

→ Copper(II) sulfate solution is toxic by ingestion, and is irritating to the skin. Handle cautiously. Do not eat or drink in lab.

→ Use caution when heating copper sulfate solution. Bunsen burner or hot plate will be hot. Use tongs to move the hot solution.

→ Follow all classroom safety procedures.

→ Wash your hands thoroughly with soap and water before leaving the laboratory.

Setup/Preparation

→ If using a filter funnel, set up a ring stand and attach the ring. Place a 100-mL beaker on the ring stand base or lab bench. Put the filter funnel in the ring, and adjust the height of the ring such that the bottom of the funnel is about an inch above the beaker.

→ If using a Buchner funnel, attach the filter flask to a suction source using plastic or rubber tubing. Insert the Buchner funnel and ensure there is a good seal.

Procedure Tips

→ Record all data immediately in the data table on page S8.

→ Lightly scratch the bottom of the 100-mL beaker to facilitate precipitation.

→ When filtering, it may be helpful to *lightly* scrape the filter paper with a rubber policeman to prevent the pores from blocking.

→ After filtering, to speed the drying process, lay the filter paper face-up on a flat surface, and spread the crystals in a thin layer across the paper.

Procedure

1. Obtain a clean, dry 100-mL beaker, and record its mass.
2. Add copper(II) sulfate pentahydrate (mass calculated in Pre-Lab) to the beaker, mass it again, and record.
3. Add 15 mL of DI water to the beaker and stir until dissolved. The solution can be gently heated if the copper(II) sulfate will not fully dissolve. Use tongs to move the hot solution. Cool the beaker to room temperature if heated.
4. In a fume hood, measure *twice* the volume of ammonia calculated in the Pre-Lab. Record the volume.
5. While in the hood, add the ammonia slowly, while stirring, to the copper(II) sulfate solution. Note any changes in color, or formation of precipitates.
6. While continuing to stir the solution, add 25 mL of ethanol to the solution. Crystallization should begin at this point. Stop stirring and allow to stand for 5 minutes.
7. The bottom of the beaker may be scratched with a glass stir rod to facilitate precipitation.
8. Obtain a piece of filter paper and record its mass.
9. If using a filter funnel, fold the filter paper in half, then in half again. Use a finger to open it to create a cone shape. Place this in the filter funnel, and use DI water to wet just enough so that the filter paper sticks to the sides.
10. If using a Buchner funnel, place filter paper in the bottom of the funnel and ensure that there are no holes visible.
11. The beaker can be removed from the fume hood at this point.
12. Slowly pour the solution into the funnel. Make certain the solution never rises above the top of the filter if using a filter funnel.
13. Use a stir rod with rubber or plastic policeman to transfer any remaining crystals to the funnel.

Procedure (continued)

14. Use a 5-mL aliquot of ethanol to rinse the beaker. Add this ethanol to the funnel, and repeat the rinse with another 5-mL aliquot of ethanol.
15. Rinse the crystals in the funnel with any remaining ethanol, in 5-mL aliquots.
16. Allow the crystals to rest in the funnel and filter for 10–15 minutes.
17. Remove filter paper and crystals from the funnel, and allow them to dry completely.
18. Once the crystals are dry, record the mass of the filter paper and crystals together.

Disposal and Cleanup

→ Dispose of all chemicals and solution as instructed by your teacher.

→ Clean all glassware and return all equipment and supplies to their proper place.

→ Wash hands thoroughly before leaving the laboratory.

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Data Table

Synthesis of a Coordination Compound

Mass of 100-mL beaker (g)	
Mass of 100-mL beaker with $CuSO_4 \cdot 5H_2O$ (g)	
Mass of $CuSO_4 \cdot 5H_2O$ (g)	
Volume of NH_3 (mL)	
Mass of filter paper (g)	
Mass of filter paper with product (g)	
Mass of final product (g)	
Observations:	

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Questions and Calculations

Synthesis of a Coordination Compound

1. Using the initial mass of $CuSO_4 \cdot 5H_2O$, determine how many moles were used.

2. Why was double the stoichiometric volume of NH_3 used in the experiment?

3. Determine the theoretical yield of ($[Cu(NH_3)_4]SO_4 \cdot H_2O$).

4. Determine percent yield using the experimental data.

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5. Why was the presence of water kept to a minimum in this reaction?

6. Identify sources of error that could have altered the yield of ($[Cu(NH_3)_4]SO_4 \cdot H_2O$).