Lab 5. Preparation of several transition metal complexes

**Introduction**

One of the most intriguing features of the transition metal ions is their vast array of colors. These colors can change when ligands bind to a metal and produce changes in the electronic energy levels of the metal ion as seen in a compound’s visible spectrum.

In this lab, you will prepare the following three coordination compounds and calculate % yields:

1. Tetraamminecopper(II) sulfate monohydrate, [Cu(NH3)4]SO4·H2O

2. Hexamminenickel(II) chloride, [Ni(NH3)6]Cl2

3. Tris(ethylenediamine)nickel(II) chloride dihydrate, [Ni(en)3]Cl2·2H2O

The preps are straightforward and require no recrystallization.

**Background to Reactions**

*1. Tetraamminecopper(II) sulfate monohydrate*

Ammonia is added to an aqueous solution of copper(II) sulfate pentahydrate. Ammonia displaces the four water ligands from the coordination sphere:

[Cu(H2O)4]SO4·H2O + 4 NH3 → [Cu(NH3)4]SO4·H2O + 4H2O Equation 1

The solution is cooled and ethanol is added to reduce the solubility of the blue tetraamminecopper (II) sulfate which precipitates from the solution.

*2. Hexaamminenickel(II) chloride*

For the preparation of hexaamminenickel(II) chloride, six ammonia molecules displace the six water ligands in [Ni(H2O)6]Cl2

[Ni(H2O)6]Cl2 + 6NH3 → [Ni(NH3)6]Cl2 + 6H2O Equation 2

The [Ni(NH3)6]Cl2 is cooled and precipitated with ethanol; the lower polarity of the ethanol causes the lavender product to become less soluble.

*3. Tris(ethylenediamine)nickel(II) chloride dihydrate*

Three ethylenediamine (en) molecules displace the six water ligands of [Ni(H2O)6]Cl2 to form violet tris(ethylenediamine)nickel(II) chloride dihydrate.

[Ni(H2O)6]Cl2 + 3 en → [Ni(en)3]Cl2·2H2O + 4 H2O Equation 3

**Experimental Procedure**

*Caution: conc HCl, conc NH3, and the ethylenediamine reagents should be used under the bench fumehoods. Wear rubber gloves when using. Place waste in labeled containers.*

**Use the small filter funnels for all three preps. Use graduated cylinders for volumn measurements. Solid reagents can be carefully weighed on the small lab balances. Clean up any spilt solid.**

**1. Synthesis of tetraamminecopper(II) sulfate monohydrate**

Weigh 1.0 g of copper(II) sulfate pentahydrate in a clean dry 25-mL Erlenmeyer flask. Dissolve the solid in 3.0 mL of deionized water. Heating may be necessary. Collect about 3 mL of concentrated ammonia and add dropwise to the copper solution (under the fumehood) until the precipitate that initially forms dissolves.

Cool the deep blue solution in an ice bath. Cool 3 mL of ethanol (in a test tube) in an ice bath and then slowly add to the solution dropwise. A blue solid complex product should form.

Collect the solid by vacuum filtration.

Wash the solid with 1-mL portion of cold ethanol followed by 1 mL of cold acetone. Allow to air dry under the vacuum for several minutes. Determine the mass of the product and store in a labeled vial.

**Questions**

Q1. When ammonia (ammonium hydroxide) is first added to the copper (II) sulfate, a pale-blue solid precipitates but dissolves forming the dark blue tetraamminecopper (II) as more ammonia is added as shown in equation 1 above. What might the pale-blue precipitate be?

Q2. Calculate the percent yield of tetraamminecopper (II) sulfate in equation 1.

Q3. The water in solid tetraamminecopper (II) sulfate is actually coordinated to the metal. Draw and name the structure of the complex (search online if needed).

Q4. The visible spectrum of

 tetraamminecopper (II) sulfate is shown at right:

 Is this what you would expect, given the color of the

 compound? Explain.

**2. Synthesis of hexaamminenickel(II) chloride**

In a 25-mL Erlenmeyer flask, dissolve 1.0 g of nickel(II) chloride hexahydrate in no more than 2.0 mL deionized water ( warm on hotplate if needed). Under the fumehood, slowly add 3.3 mL of conc NH3 (over about 30 sec)

Cool the mixture in an ice bath. Cool 2.5 mL of ethanol (in a test tube) in an ice bath then slowly add it to the solution dropwise. Allow the mixture to settle for complete precipitation of the lavender product.

Collect the solid by vacuum filtration.

Wash the solid with 1 mL of cold ethanol followed by 1 mL of cold acetone. Determine the mass of the product and store in a labeled vial.

Q5. Calculate the percent yield in equation 2.

Q5. Draw the structure of the complex and name its geometry.

Q6. What would be the approx. λmax in the visible spectrum of this compound?

**3. Synthesis of tris(ethylenediamine)nickel(II) chloride hydrate**

In a 25-mL Erlenmeyer flask, dissolve 1.0 g of nickel(II) chloride hexahydrate in 2.0 mL of deionized water (warm on hotplate if needed). Cool the mixture in an ice bath. Under the bench fumehood, slowly add 1.7 mL ethylenediamine.

Cool the mixture in an ice bath. Cool 2.5 mL ethanol (in a test tube) in an ice bath and then slowly add it to the solution dropwise. Allow the mixture to settle for complete precipitation of the lavender product.

Collect the solid by vacuum filtration.

Wash the solid with 1-mL cold ethanol followed by 1 mL of cold acetone. Determine the mass of the product and store in a labeled vial.

Q7. Calculate the percent yield in equation 3.

Q8. Draw and name the structure of the complex.