

## Synthesis of Cobalt Coordination Compounds:

### $[\text{Co}(\text{NH}_3)_5\text{Cl}]\text{Cl}_2$ , $[\text{Co}(\text{NH}_3)_5\text{ONO}]\text{Cl}_2$ , and $[\text{Co}(\text{NH}_3)_5\text{NO}_2]\text{Cl}_2$

Consult Chemistry, The Central Science, 9<sup>th</sup> edition, Brown, LeMay, Bursten, Chapter 24 for additional information about coordination compounds.

Each student will prepare one coordination compound.

**Safety Precautions: Be especially careful in handling concentrated ammonia, concentrated hydrochloric acid and 30% hydrogen peroxide!**

#### I Preparation of $[\text{Co}(\text{NH}_3)_5\text{Cl}]\text{Cl}_2$

In the fume hood, completely dissolve 6 g of  $\text{NH}_4\text{Cl}$  in ~40 mls of concentrated ammonia in a 400 ml beaker. With continuous stirring, add 12 g of cobalt(II) chloride-hexahydrate in small portions. With continued stirring of the resulting brown slurry, **SLOWLY** add 10 mls of 30%  $\text{H}_2\text{O}_2$ . After the effervescence has stopped, **SLOWLY** add ~30 mls of concentrated  $\text{HCl}$ . With continued stirring, heat on a hot plate and maintain  $85^\circ\text{C}$  for 20 minutes.

Cool mixture to room temperature in an ice bath and filter (using a Buchner funnel) the crystals of  $[\text{Co}(\text{NH}_3)_5\text{Cl}]\text{Cl}_2$

Wash with 5-6, 5 ml portions of ice water (distilled water cooled in ice) and then 5-6, 5 ml portions of cold 6 M  $\text{HCl}$ . Note the color of your product.

Dry the product in an oven at  $100^\circ\text{C}$  for several hours.

Weigh the product. Calculate the theoretical and experimental percent yield.

#### IIA Preparation of $[\text{Co}(\text{NH}_3)_5\text{ONO}]\text{Cl}_2$

In the fume hood, completely dissolve 2.5 g of  $[\text{Co}(\text{NH}_3)_5\text{Cl}]\text{Cl}_2$  in a solution of 5 mls of concentrated ammonia in 40 mls of distilled water. Stir and heat if necessary to dissolve. (A black precipitate of cobalt oxide may form.) Cool the solution to  $\sim 10^\circ\text{C}$ . Add 2 M  $\text{HCl}$  dropwise with stirring until the solution is neutral to litmus. Dissolve 2.5 g of sodium nitrite in the solution followed by 2.5 mls of 6 M  $\text{HCl}$ .

Cool mixture to room temperature in an ice bath for at least 45-50 minutes and filter (using a Buchner funnel) the precipitated crystals of  $[\text{Co}(\text{NH}_3)_5\text{ONO}]\text{Cl}_2$

Wash with 5-6, 5 ml portions of ice water (distilled water cooled in ice) and then 5-6, 5 ml portions of alcohol. Note the color of your product.

Allow the product to air dry for several hours.

Weigh the product. Calculate the theoretical and experimental percent yield.

## IIB Preparation of $[\text{Co}(\text{NH}_3)_5\text{NO}_2]\text{Cl}_2$

In the fume hood, completely dissolve 2.5 g of  $[\text{Co}(\text{NH}_3)_5\text{Cl}]\text{Cl}_2$  in a solution of 5 mls of concentrated ammonia in 40 mls of distilled water. Stir and heat if necessary to dissolve. (A black precipitate of cobalt oxide may form.) Cool the solution to  $\sim 10^\circ \text{C}$ . Add 2 M HCl dropwise with stirring until the solution is neutral to litmus. Dissolve 2.5 g of sodium nitrite in the solution followed by 2.5 mls of 6 M HCl.

Cool mixture to room temperature in an ice bath for 10 minutes and confirm that crystals are present.

Gently warm the solution and add a few drops of 6 M  $\text{NH}_4\text{OH}$ . Cool mixture and add 20 ml of concentrated HCl. Cool the solution to  $10^\circ \text{C}$  for 45-50 minutes and filter (using a Buchner funnel) the precipitated crystals of  $[\text{Co}(\text{NH}_3)_5\text{NO}_2]\text{Cl}_2$

Wash with 5-6, 5 ml portions of ice water (distilled water cooled in ice) and then 5-6, 5 ml portions of alcohol. Note the color of your product.

Allow the product to air dry for several hours.

Weigh the product. Calculate the theoretical and experimental percent yield.

Reference: The Synthesis and Characterization of Inorganic Compounds, Jolly, Prentice-Hall, 1991.

Name: \_\_\_\_\_

Preparation I IIA IIB  
(circle one)

Date: \_\_\_\_\_

Laboratory Instructor: \_\_\_\_\_

### Preparation of Transition Metal Coordination Compounds

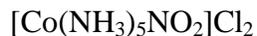
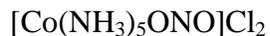
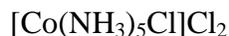
1. Color of:  
[Co(H<sub>2</sub>O)<sub>6</sub>]Cl<sub>2</sub> \_\_\_\_\_  
[Co(NH<sub>3</sub>)<sub>5</sub>Cl]Cl<sub>2</sub> \_\_\_\_\_
2. Mass of starting material (I or IIA or IIB) \_\_\_\_\_
3. Coordination compound prepared: \_\_\_\_\_
4. Chemical reactions:  
Preparation I:  
  
Preparation IIA:  
  
Preparation IIB:
5. Theoretical yield of compound prepared (show calculations):
6. Experimental yield of compound prepared: \_\_\_\_\_
7. Percent yield of compound prepared (show calculations):

8. Color of:  
[Co(NH<sub>3</sub>)<sub>5</sub>Cl]Cl<sub>2</sub> (product I) \_\_\_\_\_  
[Co(NH<sub>3</sub>)<sub>5</sub>ONO]Cl<sub>2</sub> (product IIA) \_\_\_\_\_  
[Co(NH<sub>3</sub>)<sub>5</sub>NO<sub>2</sub>]Cl<sub>2</sub> (product IIB) \_\_\_\_\_

Name: \_\_\_\_\_

**Questions:**

1. Did your synthesis produce the desired product? Cite the evidence to support your answer.
2. Draw the structure for each of the following compounds (be sure to correctly indicate the difference in metal-ligand bonding between the last two compounds):



3. Name the following compounds



4. Write the formula of the following compounds:

