**Synthesis and Characterization of Cobalt Complexes**
Adapted from “Preparation and Analysis of Two Coordination Complexes of Cobalt”, Carnegie Mellon University.

**For this experiment:**

1. Complete the Prelab and obtain a stamp **before** you begin the experiment.
2. Write your lab notebook prelab and get it initialed/signed **before** you begin the experiment. Be sure to summarize the synthetic procedure for your complex.
3. Students whose last names begin with A – Mc will synthesize Complex 1. Students whose last names begin with Me – Y will synthesize Complex 2. You will need to find someone who has synthesized the other complex and share data with them for the Report Sheet.
4. Read the section on filtration in your *Laboratory Handbook*.
5. Determine the theoretical and percent yield for your synthesis, and report this on the Data Report Sheet.
6. Develop a method using conductivity measurements to determine the chemical formula for the compound you synthesized.

**Turn in your Data Report Sheet, Stamped Prelab, and notebook pages for this experiment.**

**Introduction**

In this experiment, you will be synthesizing one of two coordination complexes consisting of cobalt, ammonia, and chloride ions. Once you have prepared the complex, you will use conductivity measurements to determine the exact formula of this compound. Finally, you will work with another student and compare results to determine the formula of the other complex.

Cobalt, a transition metal, is a rare but important element with a variety of uses. It is found in alloys of steel used as stainless steel and surgical steel. It is also used as a pigment, in magnets, and in industrial catalysts. Biologically, cobalt is found in trace amounts in the complex *cobalamin*, or Vitamin B12. This complex is part of certain coenzymes used in the synthesis of red and white blood cells and in the normal growth and maintenance of nerve cells.

Many transition metals form coordination complexes, which are ionic compounds containing a polyatomic ion that is based around the transition metal ion. The metal ion itself is positively charged and is therefore a Lewis Acid, or an electron pair acceptor. Also, since it is a single-atom ion, the charge of the metal is the same as the oxidation state. Surrounding this metal cation are some number of small molecules or ions called ligands; there are typically 2 – 9 ligands for each metal ion. These ligands are Lewis Bases, and all have a lone pair of electrons that can be donated to the metal ion. Some ligands have multiple atoms with multiple lone pairs, and each atom can attach to the metal. These ligands are called **multidentate** ligands, from the greek words meaning (loosely) “many teeth”. Several examples of monodentate and multidentate ligands are shown below.

Monodentate ligands: Cl\(^-\), NH\(_3\), H\(_2\)O, CO, CN\(^-\)

Multidentate ligands:

- ethylenediamine (en)
- phenanthroline (phen)
- catechol
- ethylenediaminetetraacetic acid (EDTA)
Many transition metal compounds form octahedral molecules, where the metal is the central atom and 6 ligands arrange themselves evenly around metal. However, if a metal can have up to 9 ligands, where do the remainder go? The ligands fall into two categories: inner sphere ligands and outer sphere ligands. Inner sphere ligands are directly covalently bonded to the metal. Outer sphere ligands are there solely as counter ions to balance the overall charge of the complex. As a result, the only ligands that can be found in the outer sphere are charged ligands. A special exception to this rule is water, which can also be an outer sphere ligand. When water is an outer sphere ligand, it is called a water of hydration. For example, let’s consider the complex \([\text{Fe}(\text{H}_2\text{O})_4\text{Cl}_2]\). Looking at the overall molecule, there are 3 Cl\(^{-}\) ions, so the oxidation state of the Fe must be +III, as water is a neutral ligand. Within the square brackets are the 6 inner-sphere ligands — 4 waters and 2 chlorides. Overall, the net charge on the complex ion is +1. To balance this +1 charge is the one outer-sphere chloride. These are listed outside of the square brackets, and are only ionically bonded to the complex. As a result of this bonding pattern, when the above complex is dissolved in water, two ions form, \([\text{Fe}(\text{H}_2\text{O})_4\text{Cl}_2]^{+1}\) and Cl\(^{-}\).

You will be synthesized one of two cobalt complexes in this experiment. Each complex contains one cobalt atom, 3 chlorides and a variable number of ammonia molecules (5 or 6). After the synthesis and recovery of your product, you will determine the exact chemical formula using conductivity measurements. Once you have determined the exact chemical formula for your compound, you will calculate the theoretical yield and percent yield of your complexes.

**PROCEDURE**

**HAZARDS**

1. 30% Hydrogen peroxide is a very caustic material! It is a very strong oxidizing agent and will readily oxidize the organic material in skin, causing severe burns. The dead skin will be “bleached” white almost immediately. Wash thoroughly with water in the event of exposure. You should also avoid peroxide contacting any combustible materials, for example clothes, paper towels and organic solvents. If you must wipe up a spill, rinse the paper towel with water in the sink before throwing it in the trash can.

2. Concentrated ammonium hydroxide is very caustic. Wash exposed areas thoroughly with water. Breathing the vapors can cause dizziness and fainting. Work with this material in a fume hood. People showing symptoms of overexposure should be removed to fresh air immediately.

3. Concentrated hydrochloric acid is very corrosive! In the event of contact, wash the area thoroughly with water. Avoid breathing the vapors of the concentrated reagent. Work in a fume hood.

4. Denatured ethanol is poisonous! Additional chemicals have been added to the ethanol that are toxic and will induce vomiting.

Weigh all materials on the analytical balances, to 0.1 mg. Be sure to write down all digits given by the balance!

All steps **in bold** must be carried out in a fume hood.

**Complex 1** (last name begins with A – Mc)

1. Weigh 6 g cobalt (II) chloride hexahydrate and set aside for step 3. Weigh out 0.5 g charcoal and place in a 250-mL Erlenmeyer flask.

2. Weigh 4 g ammonium chloride and place into a 50 mL beaker. Add 10 mL of distilled water, and heat the solution on a hot plate just to the boiling point.

3. Add the 6 g cobalt (II) chloride hexahydrate to the hot solution.

4. **Slowly** add the hot cobalt solution to the charcoal in the Erlenmeyer flask and cool the flask and contents by running tap water along the outside of the Erlenmeyer flask until warm to the touch.
5. In the fume hood, rinse the 50-mL beaker with 13 mL of concentrated ammonium hydroxide and carefully add this to the cobalt/charcoal mixture in the Erlenmeyer flask.

6. Cool the flask in an ice bath to below 10°C. Use an 800-mL or 1-L flask for this ice bath.

7. While still on ice, slowly add 16 mL 30% hydrogen peroxide to the flask. This is a VIGOROUS reaction! Add this dropwise at first, and stir after each addition, keeping the flask on ice at all times. As the reaction becomes less violent, you may increase the amount of peroxide added in each increment.

8. Place the flask in a beaker of room temperature water, and carry your mixture out of the hood and back to your bench.

9. Slowly heat your solution until it reaches approximately 60°C. Heat the solution for one hour.

10. Remove the solution from heat and cool in an ice bath to 5°C or less.

11. Filter your product using a Buchner funnel, rinsing with the filtrate. DO NOT rinse with water! To use the buchner funnels that are already set-up near the hoods, first make sure that the collection flask is empty, and clamped down. Check to make sure that the vacuum tubing from the flask is connected to the side arm/aspirator nozzle on the sink. Turn the water on at the sink, at a fairly high rate – if splashing occurs, place a beaker under the stream of water. Take a piece of filter paper and place it in the funnel, and thoroughly wet the filter paper with distilled water; this creates a seal between the filter paper and the funnel. While the paper is still wet, transfer your solution to the filter carefully, using a rubber policeman to help with the transfer. To disconnect the filtration apparatus, pull the tubing off of the flask or the aspirator tip before you turn off the water. Use your filtrate to rinse the Erlenmeyer flask for a more quantitative transfer. When you are done collecting your product, use a spatula to carefully remove the filter paper with your crude product and place it on a watch glass. Clean the buchner funnel and dispose of your filtrate and any rinses in the waste container in the hood.

**At this point, you may stop and store your product in your drawer and continue the procedure on another day. Or, you may continue with the isolation of your product now.**

12. Add 2 mL of concentrated HCl to 50 mL of water in a 250-mL beaker. Heat this solution and add the crude cobalt product to dissolve.

13. Once the crude product is dissolved, gravity filter the solution (see your laboratory handbook for technique) into a 125-mL Erlenmeyer flask. This removes the charcoal.

14. Add 8 mL of concentrated HCl to the filtrate, stir. Cool the resulting solution until it is below room temp, and solid has formed.

15. Vacuum filter your product using a buchner funnel. Rinse the 125-mL Erlenmeyer flask with 7.5 mL of denatured (95%) ethanol, and run this through the filter. Rinse your product with two more 7.5 mL portions of ethanol.

16. Transfer your product to a clean, pre-weighed watch glass, and let the solid dry overnight. Record your yield.

**Complex 2** (last name begins with Me – Y)

1. Weigh 7.5 g cobalt (II) chloride hexahydrate and set aside for step 3. Weigh 3.75 g ammonium chloride and place into a 250-mL Erlenmeyer flask. Place a stir bar in the flask.

2. In the fume hood, add 22.5 mL concentrated ammonium hydroxide to the flask.

3. With a slow rate of stirring, add 7.5 g cobalt (II) chloride hexahydrate to the flask. Stir until the cobalt is dissolved.
4. **Slowly add 6 mL 30% hydrogen peroxide. This is a VIGOROUS reaction! Add this dropwise at first, and stir after each addition. As the reaction becomes less violent, you may increase the amount of peroxide added in each increment.**

5. **After the reaction is complete and stops bubbling, add 22.5 mL concentrated HCl. Once this is added, you may take your reaction flask out of the hood.**

6. Heat, with stirring to 80-85°C. Maintain this temperature for 30 minutes.

**At this point, you may stop and store your reaction mixture, loosely covered, in your drawer. Or you may continue with the procedure.**

7. Cool the flask on an ice bath to below 10°C to completely precipitate the product.

8. Filter your product using a Buchner funnel, rinsing with water. To use the buchner funnels that are already set-up near the hoods, first make sure that the collection flask is empty, and clamped down. Check to make sure that the vacuum tubing from the flask is connected to the side arm/aspirator nozzle on the sink. Turn the water on at the sink, at a fairly high rate – if splashing occurs, place a beaker under the stream of water. Take a piece of filter paper and place it in the funnel, and thoroughly wet the filter paper with distilled water; this creates a seal between the filter paper and the funnel. While the paper is still wet, transfer your solution to the filter carefully, using a rubber policeman to help with the transfer. To disconnect the filtration apparatus, pull the tubing off of the flask or the aspirator tip before you turn off the water.

9. Rinse your flask and the solid with 3, 10-mL portions of water, followed by 3, 10-mL portions of denatured (95%) ethanol. Between washings, disconnect the vacuum and carefully stir the solid in the washing solvent, making sure to not tear the filter paper. When you are done collecting your product, use a spatula to carefully remove the filter paper with your crude product and place it on a watch glass. Clean the buchner funnel and dispose of your filtrate and any rinses in the waste container in the hood.

10. Scrape your product onto a clean, pre-weighed watch glass, and let the solid dry overnight.

**Determining the Molecular Formula**

To determine the exact molecular formula, you will use conductivity measurements to determine the number of ions in solution. To do so, you will use the conductivity meters with the Vernier lab interface, which measure conductivity in microSiemens (µS). You must determine the procedure you will use to determine the molecular formula. Available for use are 0.0100 M solutions of NaCl, Na₂CO₃, and Na₃PO₄. To use the Vernier conductivity meters, take a conductivity probe to an open computer. Make sure that the LoggerPro program is not running – quit out of the program if it is running. Plug the conductivity probe into ether LabPro box by the side of the computer, and verify that the switch on the probe is set to 0 – 20,000 µS. Open the LoggerPro program, and it will automatically start up with the correct experiment to make conductivity measurements.

Once you have determined the conductivity of your complex, find another student with data for the other complex and share your data. From these data, determine the chemical formula for both complex 1 and complex 2.
Complex ID _____

<table>
<thead>
<tr>
<th>Molecular Formula:</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Yield:</td>
<td></td>
</tr>
<tr>
<td>Theoretical yield:</td>
<td></td>
</tr>
<tr>
<td>Percent yield:</td>
<td></td>
</tr>
<tr>
<td>Characteristics:</td>
<td></td>
</tr>
</tbody>
</table>

Show your calculations for theoretical and percent yield below.

Molecular formula for the other complex: [ ]

Describe your procedure for determining the formula for your complex:
1. Define the terms inner-sphere ligand and outer-sphere ligand.

2. A nickel-ethylenediamine complex is synthesized according to the following reaction: 
   \[ \text{NiCl}_2 + 2 \text{en} + 2 \text{Cl}^- \rightarrow [\text{Ni(en)}_2\text{Cl}_2]\text{Cl}_2 \]
   You begin the reaction with 15.0 g of NiCl₂, 12.0 g of ethylenediamine, and 14.0 grams of sodium chloride. What is the theoretical yield for this reaction? 24.05 g of product is isolated. What is the percent yield for this reaction?