You may cut and paste the “Purpose” through “Skills” section. Notice that the chemical and mathematical equations and a large part of the procedure are missing.

As always, you will need to use the hints to develop your data, analysis and conclusion sections. Attach an abstract to the front of the report you turn in.

Photocopy the appropriate parts of your lab notebook; please make sure your name is legible. This lab is due Monday, March 13, in class.

Chemistry 160

Lab 7: Synthesis and Characterization of Cobalt Complexes

Purpose:
Part one: Synthesize a cobalt-based coordination compound from cobalt chloride hexahydrate.

Part two: Determine the exact molecular formula of the cobalt complex you synthesized, using conductivity measurements.

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 polydentate ligands, from the Greek words meaning (loosely) “many teeth”. Several examples of monodentate and multidentate ligands are shown below.

Monodentate ligands: Cl⁻, NH₃, H₂O, CO, CN⁻
Polydentate ligands:
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 \([\text{Fe(H}_2\text{O)}_4\text{Cl}_2]\text{Cl}\). Looking at the overall molecule, there are 3 Cl\(^-\) ions, so the oxidation state of the Fe must be +3, 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, \([\text{Fe(H}_2\text{O)}_4\text{Cl}_2]\text{Cl}^+\) and Cl\(^-\).

You will be synthesizing 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.

**Skills:**
- Performing a multi-step synthesis of a toxic and sensitive compound
- Determining the identity of a synthesized molecule
- Using a conductivity meter to test the conductivity of a substance

**Chemical equations:** Write the equation for the synthesis of your compound.

**Mathematical equation:** Percent yield equation; theoretical yield calculations

**Chemicals needed:**
- Cobalt (II) chloride hexahydrate
- Activated charcoal
- Ammonium chloride
- Concentrated ammonium hydroxide
- 30% hydrogen peroxide
- Concentrated hydrochloric acid
- 95% denatured ethanol
- ice
- 0.0100 M NaCl
• 0.0100 M Na$_2$CO$_3$
• 0.0100 M Na$_3$PO$_4$

Equipment needed:
• 250 mL Erlenmeyer flask
• 800 mL or 1 L beaker
• filter circles (Buchner + gravity)
• Buchner funnel and filtration flask
• conductivity meter
• laptop and Vernier data logger
• rubber policeman

Procedure (day one)

**Complex 1**

1. Weigh about 6 g cobalt (II) chloride hexahydrate and set aside for step 3. Weigh out about 0.5 g charcoal and place in a 250-mL Erlenmeyer flask. Of course, you should record the masses precisely.

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

3. Add the 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 beaker 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 take your mixture out of the hood and back onto 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, 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. 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 aqueous waste container.

**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 about 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 into a 125-mL Erlenmeyer flask. This removes the charcoal.

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

15. Vacuum filter your product using a Buchner funnel. Discard the filtrate in the aqueous waste. 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. Discard the filtrate in the organic waste.

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

**Complex 2**

1. Weigh about 7.5 g cobalt (II) chloride hexahydrate and set aside for step 3. Weigh about 3.75 g ammonium chloride and place into a 250-mL Erlenmeyer flask. Place a stir bar in the flask. Of course, you should record the masses precisely.

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

3. **With a slow rate of stirring, add the cobalt (II) chloride hexahydrate to the flask. Stir until the cobalt is dissolved.**

4. Slowly add about 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 about 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, 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. 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 a 10-mL portion of water. Repeat twice more and discard the filtrate into the aqueous waste. Between washings, disconnect the vacuum and carefully stir the solid in the washing solvent, making sure to not tear the filter paper. Then rinse your flask and the solid with a 10-mL portion of denatured (95%) ethanol. Repeat twice more and discard the filtrate into the organic waste. 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.

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

Procedure (part two)

The following is a general idea of how to do this part of the experiment. I expect you to write in your notebook your actual steps how you measured the conductivity AND how you calibrated that conductivity into a number of ions per molecule.

[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 the 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.]

Hazards:
• 30% hydrogen peroxide is a 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 prevent 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.

- Concentrated ammonium hydroxide is 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.

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

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

**Waste disposal:** Place all water-based waste in the “aqueous waste” container in the hood. Place all 95% ethanol waste in the “organic waste” container in the hood.

**Data section:**

Table with mass data, description of reactants, description of products

Observations of reacting mixture during various parts of the reaction

Table with conductivity data, including the control solutions

**Analysis/calculations**

Show your calculations for theoretical and percent yield.

<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>
</tbody>
</table>

Complex identity based on conductivity data.

**Conclusion**

Address the following points:

- What was the product that you synthesized? What did it look like, compared to what the reactant looked like? What was your percent yield? Is this a reasonable amount of yield to expect?

- How vigorously did the reaction occur when the hydrogen peroxide was added? Did the temperature of the solution get too high at any point during the experiment?

- What were the major sources of yield loss? Were these sources fixable and/or out of
your control? If they were fixable, how would you do this experiment next time?

Abstract

Usual format. Include product name, reactant name(s) and percent yield. Add a statement on whether the yield was acceptable, and state the major source(s) of yield loss. Describe, briefly, how the identity of the product was obtained. Comment on whether this method of identification gave unequivocal results.