Experiment 7: Isolation of cis- and trans- Isomers of a Cobalt (III) Compound
CH3500: Inorganic Chemistry, Plymouth State University


Introduction:

Isomers are molecules that have the same chemical formula but different structure. Several different types of isomers are possible, depending on how the atoms are arranged and what makes two related isomers different (Figure 1).

![Figure 1: Types and characteristics of Isomers](image)

Diastereomers, especially the cis/trans variety, are common isomers encountered in organic chemistry, and they are also found in inorganic chemistry. The terms "cis" and "trans" mean roughly "next to" and "across from" in the same way they do in organic chemistry, though they are encountered in inorganic coordination compounds in a slightly different way (Figure 2).

![Figure 2: cis and trans isomers in a) organic molecules and b) inorganic coordination compounds](image)

The physical and chemical properties of cis and trans isomers, including color, melting and boiling point, stability, and chemical reactivity, can be very different from one another. In this lab, you will synthesize both the cis and trans isomers of dicholorobis(ethylenediamine) cobalt(III) [Co(en)₂Cl₂]⁺. The trans isomer is more thermally stable, especially in solution, and is isolated first. A portion of the trans isomer is converted into the cis isomer and isolated by selective precipitation.
Safety Considerations:
- Do NOT dispense volatile organic liquids outside the hood.
- Reactions should be run in a fume hood.
- Be careful when heating solutions of strong acids!
- Properly dispose of organic solvents (e.g. ether) in the Organic Waste container.

Procedure
- You will work with a partner for this lab. You may wish to divide up the tasks!
- Remember to record what you do (including exact masses weighted out), data collected, and observations at each step in the procedure.

A. Preparation of trans-[Co(en)$_2$Cl$_2$]Cl
1. Start heating a beaker containing about 300 mL of water on a hotplate. The heat should be set so that the water boils gently, not vigorously.
2. Dissolve 1.5 g of cobalt(II) chloride hexahydrate in 10 mL of distilled water in a large vacuum-flask-style test tube.
3. Add 5 mL 10% ethylenediamine to the test tube.
4. Use a marker to draw two short lines indicating approximately half and one third the volume in the test tube.
5. Along with a second large vacuum-flask-style test tube and the necessary tubing, stoppers, and glass tubes, setup a reaction apparatus as shown in Figure 3. If you are using a vacuum aspirator instead of a vacuum pump, you may omit the vapor trap. Use a ring stand and clamps to hold the test tubes in place. Do NOT start the vacuum yet!
6. The tube to the vacuum should have a glass valve connection in the middle. Make sure this valve is completely closed. Turn on the vacuum and SLOWLY open the valve until air begins to bubble gently through your solution. BE CAREFUL not to bubble air so vigorously that your solutions gets sucked into the vacuum!!
7. Allow the reaction to aerate in the boiling water bath for 1 hour. During this time, the volume of the reaction will reduce due to evaporation. Periodically check, and if the volume has fallen below the bottom line (one third the initial volume) you drew in Step 4, add a small amount of water. Ideally, the volume will be between the two lines you drew at the end of one hour.
8. At the end of 1 hour, remove the test tube from the hot water bath and immediately add 5 mL concentrated HCl.
9. Transfer the solution to an evaporating dish. Place the dish over your boiling hot water bath to reduce the volume. When small crystals begin to form, remove from the heat, add 1 mL concentrated HCl, and transfer the mixture to a small beaker.
10. Allow the mixture to cool for a few minutes at room temperature. You should see crystals beginning to form. If not, return to Step 9. After a few minutes, place the beaker in ice.
11. Filter the crystals under vacuum using a small, clean vacuum flask and a fritted funnel. Rinse the crystals with less than 1 mL ethanol and dry them with the vacuum on for a few minutes. If this was the first time you filtered, do step 'a' below. If the second time, do step 'b'.
   a) First filtration: Transfer the green crystalline powder to a small, weighed beaker and set aside. Transfer the filtrate from the vacuum flask to an evaporating dish and return to Step 9.
   b) Second filtration: Combine the crystals with those from step 'a'. Dispose of the filtrate in the proper waste container.

12. Add 5 mL methanol to the beaker with the green crystals. Using a glass stirring rod or a small spatula, stir the slurry well, being sure to break up any clumps.

13. Place the beaker in the drying oven at 105 °C for one hour.

14. Weigh the beaker to get the final mass of trans isomer. Transfer the solid to a labeled vial.

15. Obtain the UV-Vis spectrum of the trans isomer in water.

B. Preparation of \textit{cis-}[Co(en)\textsubscript{2}Cl\textsubscript{2}]Cl

\textit{You will be given pre-made trans-isomer to carry out this step, so that you and your partner may work on both parts simultaneously}

1. Place 0.2 g trans-isomer in a small evaporating dish and add 5 mL of water.
2. Heat the dish over a steam bath to evaporate the water to dryness.
3. Scrape the solid onto a micro-filtration funnel. Set up the funnel for vacuum filtration, but leave the vacuum off.
4. Add 3 drops of chilled water, stir for 20 seconds, then turn on the suction. A violet solid should remain on the filter, while a saturated green filtrate will be filtered through.
5. Transfer the filtrate back to the evaporating dish and return it to the steam bath as in Step 2.
6. Rinse the violet precipitate with 3 mL ether. Repeat, and then discard the rinse. Transfer the solid to a weighed vial.
7. When the filtrate from Step 5 has again evaporated to dryness, repeat Step 4.
8. You may repeat the dry-wash-filter process a total of three or four times to obtain the maximum amount of cis-isomer. Collect all of the violet crystals together in the same vial. Weigh the vial in the end to get the final mass of crystals.
9. Obtain the UV-Vis spectrum of the \textit{cis} isomer in water.

\textbf{Analysis (Lab Notebook)}

1. Calculate the percent yield of both compounds.

\textbf{Analysis and Conclusions (Lab Report)}

Your lab report is due by lecture on Wednesday, . Your report must be handed in BOTH electronically (via Moodle) and in hard copy form. See the document "InorgChem-LabReportGuide.pdf" on the course website (http://oz.plymouth.edu/~jsduncan/courses/2010_Fall/InorganicChemistry) for guidelines on writing your report.

1. Include in your report drawing of the cis and trans isomers of dichlororobis(ethylenediamine) cobalt(III) drawn in using a chemical drawing software package.
2. What are the point groups for the two isomers?