Experimental

A1. Synthesis of *trans*-dichlorobis (ethylenediamine) cobalt(III) chloride

In the first part of a two-part experiment, you will synthesize and isolate a Cobalt(III) coordination complex. In the next lab period you will perform a kinetic analysis of the aquation of this complex.

The balanced equation for the synthesis of this coordination complex is:

$$2Co^{2+} + H_2O_2 + 2H^+ + 4en + 4Cl^- \rightarrow 2[Co(en)_2Cl_2]^+ + 2H_2O \qquad (14)$$

(Ethylenediamine, abbreviated as "en", is $H_2NCH_2CH_2NH_2$.) The synthesis works well if the directions are followed closely; the equilibria involved are complicated, resulting in the formation of various competing products if the procedure is not followed carefully. For instance, if too much ethylenediamine is added, the yellow tris-(ethylenediamine) cobalt(III) may form; another common side product is the purple cis-dichlorobis-ethylenediamine) cobalt(III).

Procedure

The following synthesis should be carried out in a fume hood. The cooling and filtration steps can be carried out on the bench top.

Synthesis

Dissolve 4.00 grams of $CoCl_2 \cdot 6H_2O$ in 32 mL of distilled water in a 250 mL Erlenmeyer flask. *With stirring,* add 16 mL (in 2-3 mL portions, waiting 2 minutes between additions) of a 10% ethylenediamine solution slowly. After the addition of the ethylenediamine is complete, stir the reaction mixture for an additional ten minutes. Add in 1-mL portions, 5 mL of a solution of 7.5 % H_2O_2 and stir the resulting mixture for fifteen minutes. Finally, slowly add 10 mL of concentrated HCl to the reaction mixture. With continuous stirring, adjust the heat on the hotplate and bring the mixture to a boil and, in a controlled fashion, concentrate the reaction mixture to a volume of 10 mL. To avoid spattering, the temperature of the hotplate should be reduced as you approach the final volume. Remove the flask from the hot plate and allow it to cool to room temperature by placing it on a folded towel on the bench top. After it has reached room temperature, the flask may be placed in a cold water bath. Once the solution has cooled thoroughly, the crystals should be collected by suction filtration – use methanol to help transfer the product to the Buchner funnel.

Filtration

Prepare a Büchner funnel setup according to the figure below and the directions given by the laboratory instructor. The various components of the apparatus should be securely clamped before any chemical manipulations are begun. The filter paper should be small enough to fit **flat** on the funnel but large enough to cover all of the holes. Wet the paper with a little water and attach the trap, and turn on the suction before pouring the slurry onto the filter paper.

With suction on, swirl the reaction flask to suspend the precipitate and transfer the reaction mixture to the funnel in one operation (checking first to see that the volume of reaction mixture does not exceed the volume of the funnel). After you have drained the reaction flask and the supernatant liquid has been drawn through the Büchner funnel, add a 2-mL portion of chilled methanol to wash down the sides of the flask. Swirl to suspend the remaining precipitate and transfer again to the funnel. Repeat this washing with one additional 2-mL portion of cold methanol.

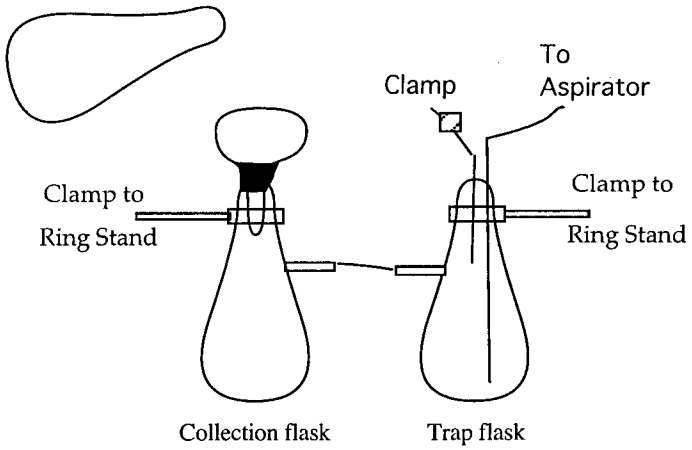

Figure 1. Setup for a vacuum filtration. Be sure to release the clamp at the top of the trap to let air in before turning off the suction.

Transfer the crystals to a clean, weighed, weighing bottle, and place in a dessicator. You should plan to return to lab after 48 hours (but before your next lab) to weigh the dried crystals and calculate a percentage yield.

Clean the collection flask and return it to the lab.