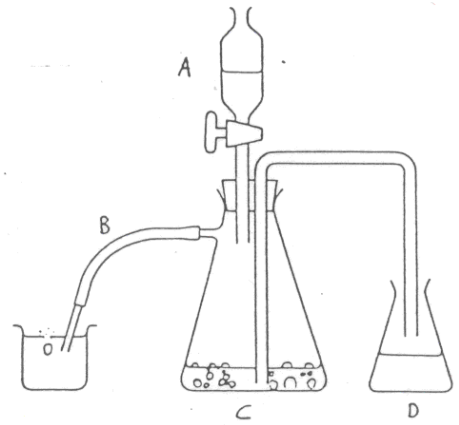
Chem 151L, Spring 2009
Expt. 4. Preparation of Chromium(II) Acetate

Chromium(II) acetate, or chromous acetate, is perhaps the most accessible representative of the large and growing class of complexes with an unusual type of metallic bonding. A major point of this lab exercise will be not only the challenging preparation of this complex, but further study of the understanding it has brought to bonding theory. You will work in a relatively oxygen free environment and study the following oxidation-reduction couple:

$$Cr^{3+} + e^- \rightarrow Cr^{2+}, \quad E^o = -0.41 \text{ V} \qquad [1]$$



Place 6 g of mossy zinc and a solution of 7.5 g of $CrCl_3 \cdot H_2O$ dissolved in 25 mL of water into flask C (see figure). Set up the apparatus securely so that the side-arm tube B is just below the surface of the water in the beaker. **Slowly and with care**, add concentrated HCl to the reaction solution from dropping funnel A until the solution becomes a permanent, clear blue color (~ 12.5 mL). **Note:** take care during the addition so that no solution rises into the transfer tube leading to flask D.

Solution D is prepared by adding 45 g of sodium acetate to 40 mL of water in a 125 mL erlenmeyer flask. This concentrated solution should be warmed until it is clear and bubble free. At this point, the flask should be closed with a stopper that is connected to a balloon that contains ~ 125 mL of N_2 gas. When cooled, the flask should be fitted to the transfer arm and sealed with parafilm.

With the production of the blue solution, transfer can be effected by carefully pinching tube B. During transfer, raise D so that the tube is just under the surface of the solution. Swirl D for good mixing. A drop or two of additional HCl may be necessary for complete transfer (takes 10 to 20 minutes), or use an N_2 line to push the solution out. When complete, quickly separate and re-stopper D with the nitrogen balloon.

Collecting the crystals without oxidation by air is the hardest part of the procedure. Using a buchner funnel, rinse the solid with four 25 mL portions of ice-cold, air-free water. Never let the crystals lose a covering of rinse water. After the water, rinse with three small portions (enough to submerge the crystals) of 95% ethanol and then finally with ether. With the last portion of ether, be ready to place an inverted funnel with N_2 flowing over the buchner funnel for one minute. This will allow the crystals to warm up under a blanket of N_2. Be ready to transfer the solid into a pre-tared N_2 filled test tube and stopper tightly. Weigh and calculate your yield.

Questions:

1) At the time of the transfer of the blue solution, what is your estimate of its pH?

2) What other reducing agents would be suitable? Write reactions and standard potentials for each.

3) What is the by-product gas produced in this experiment? What special precautions must be used?

4) If the red color is lost, what has happened?

5) Why do the Zn and $CrCl_3$ not react directly in the first step?

References

1) Ocone, L. R.; Block, B. P. *Inorg. Syntheses*, **1966**, *8*, 125-131.

2) Cotton, F. A. Rice, G. W. *Inorg. Chem.* **1978**, *17*, 2004-2009.