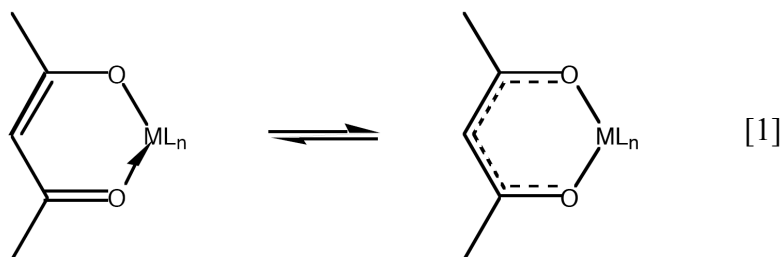


Chem 151L, Spring 2009

Expt. 3. Synthesis of Vanadium(IV) Oxy(acetylacetonate)

Vanadium coordination compounds are stable in a range of oxidation states (-1 to +5). Vanadium (IV) is of considerable interest since it is the most stable, with the metal in a d^1 configuration. Acetylacetonate (acac) is a common monovalent bidentate ligand derived from the monodeprotonation of acetylacetone (2,4-pentanedione). The binding of this ligand to a transition metal ion can be thought of as consisting of a covalent bond through one oxygen and a dative bond through the other oxygen (bond order 1.5). In reality, the bonding is delocalized and most correctly drawn using the resonance structure shown on the right (Scheme 1).



Note: this procedure produces a strong odor and must be performed in the fumehood. Add 15 mL distilled water to 5 g vanadium(V) oxide in a 400 mL beaker, then 9 mL conc. sulfuric acid (**care!**) and 25 mL ethanol. Boil the mixture on a hot plate and stir continuously. As the reaction proceeds, the initial slurry of vanadium(V) oxide darkens, becomes light green and finally turns deep blue. The time needed for V_2O_5 to dissolve and reduce to vanadium(IV) is usually 30 min. Filter and return the filtrate to a 400 mL beaker. Add 13 mL acetylacetone and neutralize the mixture by adding (slowly) a solution of 20 g sodium carbonate dissolved in 125 mL distilled water. Add the sodium carbonate solution slowly to avoid excess frothing. The product begins to precipitate at pH 3.5 and is complete at pH 5.5. Past the end point, addition of more sodium carbonate solution causes no frothing. Remove the precipitated product by filtration on a Büchner funnel and dry by drawing air through the filter cake. The yield of crude vanadium(IV) oxy(acetylacetonate) is 12 g. Recrystallize the product by first dissolving in chloroform (enough to dissolve the entire product). Heat in the fumehood until the solvent boils and wait until the product crystallizes (do not boil away the entire solvent! Remove from heat when once the crystallization appears to be complete). Let the solution cool, then filter, dry and weigh the crystals.

Questions:

- 1) What is the IUPAC name for the compound?
- 2) What is the role of ethanol in the first part of the synthesis?
- 3) Write a balanced set of equations for the synthetic steps.
- 4) What is the role of the Na_2CO_3 ?