M.Sc. II Semester

DSE-1: CYPBLD2- Inorganic Chemistry Practical-II

Synthesis of Hexamminecobalt(III) chloride $[Co(NH_3)_6]Cl_3$

Reagents:

(a) Cobalt (II) chloride hexahydrate ($CoCl_2.6H_2O$)
(b) Ammonium chloride
(c) Conc. Ammonia
(d) 30% hydrogen peroxide (H_2O_2)
(e) Conc. HCl
(f) 95% Ethanol (C_2H_5OH)
(g) Active charcoal
(h) Ice bath

Procedure:

In a 250 *ml* conical flask, dissolve ~3.5 *g* (65.5 *m.mol*) of NH_4Cl and 5 *g* (~21 *m.mol*) of $CoCl_2.6H_2O$ in ~30 *ml* of water. To the resulting solution add 1 *g* of activated charcoal and then 45 *ml* (675 *m.mol*) of conc. ammonia. Place the flask with it's contents in an ice-bath under **FUME HOOD** and cool the resulting brown slurry to 0°C (*use a thermometer*). To the ice-cool slurry, add drop wise with great **CAUTON** and continuous stirring, 4 *ml* (~35.3 *m.mol*) of 30% H_2O_2 (**CORROSSIVE**), at a rate of ~1-2 drops per second. During addition of H_2O_2, temperature of the reaction mixtures with brisk effervescences (O_2). Addition of H_2O_2 should be so adjusted that the temperature does not rise above 10°C.

After the addition of H_2O_2 is complete, remove the reaction flask from the ice-bath. Place it on an asbestos board or on a heating mantle and heat of the resulting red brown solution to ~55-60°C for ~30 minutes with occasional shaking to ensure complete transformation of the Co^{II}-aqua complex to the Co^{III}-ammine complex. Then cool the reaction mixture again to 0°C in the ice-bath. When the product crystallizes out from the solution, collect the mixture of the product and charcoal by filtration under suction.

Transfer the solid into the original conical flask with the aid of ~40 ml of hot water, add 1 ml of conc. HCl and heat the mixture to ~60-70°C, when the complex passes into the solution. Filter the mixture while still hot, cool the filtrate in the ice-bath add ~1 ml of ice-cold conc. HCl and mix uniformly. The product separates as orange-yellow crystalline solid. Collect the product by filtration under suction, wash with ~25 ml of 95% ethanol (adding in three portions of ~8 ml at a time). Air dry.

Reactions: $H_2O_2 + 2e \rightarrow 2OH^-$

$$2NH_4Cl + 2OH^- \rightarrow 2NH_3 + 2Cl^- + 2H_2O$$

$$2CoCl_2 \rightarrow 2Co^{3+} + 4Cl^- + 2e$$

$$2CoCl_2 + 2NH_4Cl + 10NH_3 + H_2O_2 \rightarrow 2[Co(NH_3)_6]Cl_3 + 2H_2O$$