

# Lab report



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Example lab report of Synthesis of potassium tris (oxalato) ferrate (III)

trihydrate Posted by Nurul Yunaliyana Experiment 5: Synthesis of potassium

tris (oxalato) ferrate (III) trihydrate Purpose: to synthesis potassium tris

(oxalato) ferrate (III) trihydrate ,  $K_3 [Fe (C_2O_4)_3] \cdot 3H_2O$ . Introduction:

Ferrous ammonium sulfate,  $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$  is dissolved in a slightly

acid solution, excess oxalic acid,  $H_2C_2O_4$ , is added and the following

reaction takes place:  $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O + H_2C_2O_4 \rightarrow FeC_2O_4(s) + H_2SO_4$

+  $(NH_4)_2SO_4 + 6H_2O$   $FeC_2O_4$  is finely divided precipitate and tends to be

colloidal.

However, heating the solution causes it to coagulate and facilitates

separating the precipitate from the solution. Potassium oxalate is added to

the  $FeC_2O_4$  precipitate, which produces a slightly basic solution for the

oxidation of the ferrous ion to the ferric ion, by hydroxide,  $H_2O_2$ . The

following reaction takes place:  $H_2O + HO_2^- + 2Fe^{2+} \rightarrow 2Fe^{3+} + 3OH^-$  The  $OH^-$

ion concentration of the solution is high enough so that some of the  $Fe^{3+}$

reacts with  $OH^-$  to form ferric hydroxide (brown precipitate) as follows:  $Fe^{3+}$

+  $3OH^- \rightarrow Fe(OH)_3$  With the addition of more  $H_2C_2O_4$ , the  $Fe(OH)_3$  dissolves

and the soluble complex  $K_3[Fe(C_2O_4)_3] \cdot 3H_2O$  is formed according to :

$3K_2C_2O_4 + 2Fe(OH)_3 + 3H_2C_2O_4 \rightarrow 2K_3[Fe(C_2O_4)_3] \cdot 3H_2O + 3H_2O$  Ethanol is

added to the solution to cause the complex iron salt to precipitate. Data

analysis and Discussion: In this experiment, I have studied how to synthesis

coordination compound. Coordination compounds are formed when a neutral

metal atom: Fe acting as a Lewis acid, reacts with some neutral molecules,

acting as Lewis bases; or when a metallic cation, acting as a Lewis acid,

reacts with any of a variety of organic or inorganic molecules, cations, or anions, acting as Lewis bases.

These Lewis bases:  $\text{C}_2\text{O}_4$  and  $\text{H}_2\text{O}$  are called ligands. (Lewis acids are electron pair acceptors and Lewis bases are electron pair donors. Ferrous ammonium solution is added with oxalic acid dihydrate solution will form yellow solution with yellow precipitate.  $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O} + \text{H}_2\text{C}_2\text{O}_4 \rightarrow \text{FeC}_2\text{O}_4(\text{s}) + \text{H}_2\text{SO}_4 + (\text{NH}_4)_2\text{SO}_4 + 6\text{H}_2\text{O}$  Then it is heated to boiling and the supernatant is decanted. As it is added with solid potassium oxalate, it is allowed to heat at  $40\text{ }^\circ\text{C}$  and drop wise added with  $\text{H}_2\text{O}_2$  and the solution turns to brown with precipitate for the oxidation of the ferrous ion to the ferric ion.

$\text{H}_2\text{O} + \text{HO}_2^- + 2\text{Fe}^{2+} \rightarrow 2\text{Fe}^{3+} + 3\text{OH}^-$   $\text{Fe}^{3+} + 3\text{OH}^- \rightarrow \text{Fe}(\text{OH})_3$  Next, more oxalic acid dihydrate is added until the solution turns to colourless.  $3\text{K}_2\text{C}_2\text{O}_4 + 2\text{Fe}(\text{OH})_3 + 3\text{H}_2\text{C}_2\text{O}_4 \rightarrow 2\text{K}_3[\text{Fe}(\text{C}_2\text{O}_4)_3] + 3\text{H}_2\text{O} + 3\text{H}_2\text{O}$  The colourless solution is boiled then it turns to pale green solution. The solution is filtered then leaves for crystallization. After that, the green crystal is filtered and washed with 1: 1 ethanol/ water and cooled acetone. The mass of bright (luminescent) green crystals is obtained which is 3. 2822 g. So, the percent yield of  $\text{K}_3[\text{Fe}(\text{C}_2\text{O}_4)_3] \cdot \text{H}_2\text{O}$  that I have obtained is 47. 72 %. The precautions that we must take are while heat the solution of ferrous ammonium sulfate and solution of oxalic acid dihydrate as it will bump. Next, beware of temperature (at least  $40\text{ }^\circ\text{C}$ ) of solution when add  $\text{H}_2\text{O}_2$  into the solution. The next experiment is determination of the percentage of ligands in coordination compounds. Conclusion : I have studied how to synthesis coordination compound which is potassium tris (oxalato) ferrate (III)

trihydrate,  $K_3[Fe(C_2O_4)_3] \cdot 3H_2O$ . The mass of bright (luminescent) green crystals is obtained which is 3.2822 g. So, the percent yield of  $K_3[Fe(C_2O_4)_3] \cdot 3H_2O$  that I have obtained is 47.72%.

## Reference:

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