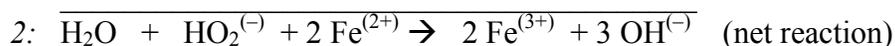
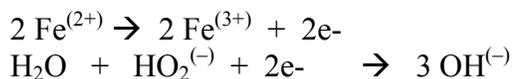

Inorganic Complex Synthesis: Preparation of Potassium tris(oxalato) iron(III) trihydrate

To prepare potassium tris(oxalato) iron(III) trihydrate, $\text{K}_3[\text{Fe}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}$, ferrous ammonium sulfate hexahydrate, is dissolved in a slightly acidic solution, excess oxalic acid is added, and the following reaction takes place:



Ferrous oxalate, FeC_2O_4 , is a 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 ferrous oxalate precipitate that produces a slightly basic solution for the oxidation of the ferrous ion to the ferric ion by hydrogen peroxide. The following reaction takes place:



Note that the ferrous oxalate is the source of the $\text{Fe}^{(2+)}$ in Equation 2. The hydroxide ion concentration of the solution is high enough so that some of the $\text{Fe}^{(3+)}$ reacts with hydroxide to form ferric hydroxide (brown precipitate) as follows:



With the addition of more oxalic acid, the ferric hydroxide dissolves and the soluble complex is formed:



Alcohol is added to the solution to cause the complex iron salt to precipitate since it is less soluble in alcohol than in water.

PROCEDURE

In a 125-mL Erlenmeyer flask, 13mmol of ferrous ammonium sulfate hexahydrate is dissolved in 15mL distilled water. The solution is acidified by the addition of several drops of dilute sulfuric acid. To this solution, add 50mL of 0.5M oxalic acid (CAUTION) and heat to boiling while stirring constantly to prevent bumping. Remove from heat and allow the ferrous oxalate to settle and decant the solid product from the liquid. Wash the precipitate twice with 20mL hot water, decanting the supernatant liquid into a waste container.

Add 20mL of 1M potassium oxalate (CAUTION) to the flask containing the precipitate, stir and heat to 40°C. As soon as the temperature is at 40°C, begin dropwise addition of

20mL of 3% hydrogen peroxide, stirring continuously. Periodically check the temperature of the solution and make sure that it is at least 40°C (but not >50°C) during the addition.

After addition is complete, heat the resulting solution to boiling. Obtain 16mL of 0.5M oxalic acid solution, add 8 mL all at once to the boiling solution. Stir continuously and then continue to add the remaining 0.5M oxalic acid solution dropwise while maintaining the temperature near boiling. The solution should turn clear green. If some brown residue remains, add an additional 1mL of 0.5M oxalic acid solution dropwise, again while the solution is boiling, until the solution is clear green. However, if the residue that remains is yellow, it is probably unreacted ferrous oxalate.

If the solution is cloudy, gravity filter it into a clean 125-mL Erlenmeyer. If it is clear, no filtration is necessary. Next, while swirling *constantly*, slowly add 15mL ethanol to the solution. Allow the solution to cool at room temperature then in an ice water bath. After crystals begin to form allow the solution to stand in the ice bath for 20 minutes. A good crop of crystals should have formed before the product is obtained by vacuum filtration. Allow crystals to air dry for about 2 minutes on the funnel, stop the suction and add 10mL of a 1:1 ethanol/water solution. Wait about 30 seconds and then apply suction for 2 minutes. Repeat this washing process. After this final wash, allow the suction to continue for a further two minutes.

Set aside to dry and obtain mass of product.

Calculate percentage yield, atom economy and e-factor.