

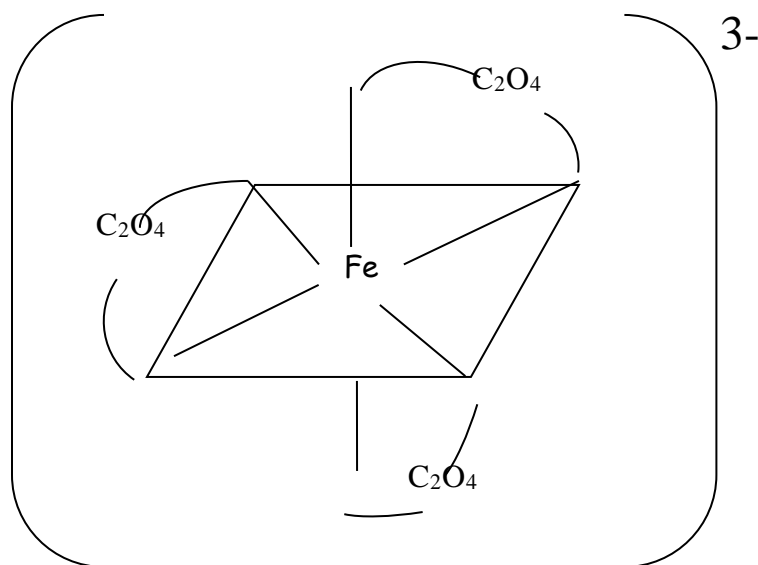
## Chemistry 112

### PREPARATION & ANALYSIS OF AN IRON COORDINATION COMPOUND PART A: PREPARATION OF AN IRON COORDINATION COMPOUND

#### A. INTRODUCTION

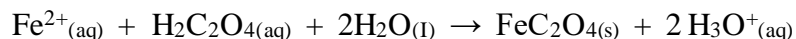
In this experiment you will synthesize the iron coordination compound, potassium trioxalatoferrate (III),  $K_3[Fe(C_2O_4)_3]$ . In the subsequent experiment, you will analyze this salt for iron (III) and oxalate ion content in order to assess its purity.

The interesting portion of the compound is the complex ion,  $[Fe(C_2O_4)_3]^{3-}$ , which has an octahedral geometry in which two oxygens from each oxalate ion occupy adjacent corners of an octahedron, with the  $Fe^{3+}$  at the center.



**Trioxalatoferrate (III) Ion**

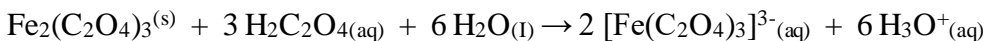
The synthesis of the salt will be carried out in several steps. In the first,  $Fe^{2+}$  ion in a solution of ammonium iron (II) sulfate,  $Fe(NH_4)_2(SO_4)_2$ , reacts with oxalic acid to form iron (II) oxalate,  $FeC_2O_4$ .



The iron (II) oxalate,  $FeC_2O_4$ , precipitate is separated from the solution and then oxidized to iron (III) oxalate,  $Fe_2(C_2O_4)_3$ , using hydrogen peroxide as the oxidizing agent and potassium oxalate as the source of oxalate ions. The net ionic equation for the reaction is:

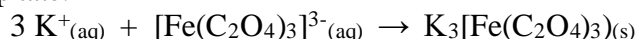


After removing excess hydrogen peroxide by heating, the iron (III) oxalate is converted to trioxalatoferrate (III) ion by treatment with oxalic acid according to the net ionic equation:



Since the salt produced,  $\text{K}_3[\text{Fe}(\text{C}_2\text{O}_4)_3]$ , is soluble in water, it will not precipitate from an aqueous solution. (The potassium ions of the salt are spectator ions in reaction 3.)

However, when ethanol (a miscible liquid) is added to the solution the resulting solvent system of water-ethanol is less polar than water. This reduction in the polarity of the solvent system, coupled with cooling of the mixture and allowing it to stand overnight, causes the salt to precipitate.



### **Safety:**

**Oxalic acid:** Poison! Danger! May be fatal if swallowed. Corrosive. Causes severe irritation and burns to skin, eyes, and respiratory tract. Harmful if inhaled or absorbed through skin. May cause kidney damage

**3% hydrogen peroxide:** May be harmful if swallowed. Causes serious eye irritation

**Dilute hydrochloric acid (HCl) and sulfuric acid (H<sub>2</sub>SO<sub>4</sub>):** can harm eyes, skin, and clothing. Handle with care. Any acid spilled on the skin or splashed into your eyes must be rinsed with a large volume of water

**Ethanol:** is Flammable. Hazardous in case of eye contact (irritant), of inhalation. Slightly hazardous in case of skin contact, of ingestion.

**Acetone:** Extremely flammable liquid and vapor. Vapor may cause flash fire. Harmful if swallowed or inhaled. Causes irritation to skin, eyes, and respiratory tract.

## **B. PROCEDURE**

### **1. FORMATION OF IRON (II) OXALATE FROM AMMONIUM IRON (II) SULFATE AND OXALIC ACID**

Weigh out approximately 7.5 g of solid ammonium iron (II) sulfate,  $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2$ , into a 250 mL beaker. Record the mass of the sample in your lab notebook. Add 20 mL of warm deionized water and 8 drops of 3 M  $\text{H}_2\text{SO}_4$  solution and stir to dissolve the solid. To the resulting solution add 35 mL of 1.0 M  $\text{H}_2\text{C}_2\text{O}_4$  solution, and heat to boiling over a Bunsen burner, stirring continuously. The solid formed is iron (II) oxalate,  $\text{FeC}_2\text{O}_4$ . Allow the solid  $\text{FeC}_2\text{O}_4$  to settle and then decant off the supernatant solution. Dispose of the supernatant by pouring it into the waste container provided.

Wash the precipitate by adding about 30mL of deionized water, warming for five (5) minutes while stirring, allowing the precipitate to settle, and then decanting

the supernatant solution. Again, dispose of the supernatant by pouring it into the waste container provided.

## **2. FORMATION OF IRON (III) OXALATE BY TREATING IRON (II) OXALATE WITH HYDROGEN PEROXIDE AND POTASSIUM OXALATE**

To the  $\text{FeC}_2\text{O}_4$  precipitate add 15 mL of saturated  $\text{K}_2\text{C}_2\text{O}_4$  solution and heat to about  $40^\circ\text{C}$ . Slowly add 30 mL of 3-5%  $\text{H}_2\text{O}_2$  solution to the mixture, stirring continuously and keeping the temperature of the mixture near  $40^\circ\text{C}$ .

## **3. Formation of trioxalatoferrate (III) ion by treating iron (III) oxalate with oxalic acid**

After the  $\text{H}_2\text{O}_2$  addition, heat the mixture to just boiling ( $40^\circ\text{C}$ ), and keep it at boiling while adding 12 mL of 1.0 M  $\text{H}_2\text{C}_2\text{O}_4$  solution – the first 10 mL all at once and the last 2 mL drop-by-drop.

At this point, the solution should be clear. If the mixture is *very* turbid, you'll need to begin the synthesis again.

## **4. Precipitation of potassium trioxalatoferrate (III) \***

Allow the solution to cool partially (still warm but not hot) and then add 8 mL of 95% ethanol.

NOTE: If the ethanol is added while the solution is still hot, most of the ethanol will evaporate.

Optional: Tie a short piece of cotton thread to a wooden splint and suspend the thread in the solution. Cover the beaker with a cover glass and allow it to stand in your locker until the next laboratory period.

Note: Potassium trioxalatoferrate (III) decomposes slowly in light, therefore, extended periods of exposure to light should be avoided as much as possible.

## **5. Isolation of potassium trioxalatoferrate (III)**

Obtain a Buchner funnel and filtering flask from the side of the room. Place a piece of filter paper in the funnel. Take your crystal mixture and filtration apparatus, as well as 5 mL of 50% ethanol and a few mL of acetone. Attach the filtration apparatus to the vacuum line. Swirl the crystal mixture and then pour it into the funnel. Wash the crystals first with 5 mL of 50% ethanol and then with a few mL of acetone. If there are crystals left in the beaker, rinse them out into the funnel with the 5 mL of ethanol.

Use tape to attach a piece of filter paper to your watch glass. Scrape your crystals onto the dry filter papers and allow the crystals to dry in the hood until the ethanol and acetone odors are gone. It may take the crystals 20 minutes or more to dry.

#### **6. Weighing of potassium trioxalatoferrate (III) crystals**

While waiting for your crystals to dry, weigh a clean, dry test tube with a cork in it. When dry, transfer your crystals to the test tube, cork it, and weigh. Record a description of your crystals in your Lab Book.

### **C. DATA AND OBSERVATIONS – Leave 5 inches**

### **D. CALCULATIONS**

#### **1. Theoretical yield of potassium trioxalatoferrate (III)**

Calculate the theoretical yield of  $\text{K}_3[\text{Fe}(\text{C}_2\text{O}_4)_3]$  using the recorded starting mass of ammonium iron (II) sulfate,  $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2$ . The ammonium iron (II) sulfate you used was the hexahydrate –  $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6 \text{H}_2\text{O}$  – which has a molar mass of 392.14 g/mole. The product is also a hydrate -  $\text{K}_3[\text{Fe}(\text{C}_2\text{O}_4)_3] \cdot 3 \text{H}_2\text{O}$  – and has a molar mass of 491.24 g/mole.

#### **2. Percent yield of potassium trioxalatoferrate (III)**

Calculate the percent yield of potassium trioxalatoferrate (III) using the theoretical yield calculated above, and the experimental (actual) yield.

## **E. QUESTIONS**

1. For what reason was 95% ethanol added in Step 4 of the Procedure?
2. State three *unavoidable* procedural errors that you believe might be the reasons why you would not obtain a 100% a yield. (Do not include any that are the result of clumsiness, such as spilling, dropping, etc.) Assess the reason impact of each on the percent yield – that is –what was the effect on the percent yield. Your discussion should be related to your percent yield.

## **F. SUMMARY**