PART B:

ANALYSIS OF IRON COORDINATION COMPOUND

In this experiment the student will perform two independently analyses by titration with potassium permanganate solution to determine the iron and oxalate ion content of the $K_3[Fe(C_2O_4)_3]\ 3H_2O$ compound and will therefore provide both a verification of the composition of the compound and as assessment of its purity.

Part A. Analysis for Oxalate Ion

In this determination, the iron coordination compound is titrated directly with standard potassium permanganate in acid solution. The permanganate oxidizes the oxalate according to the equation:

$$2MnO_4^- + 5C_2O_4^{-2} + 16H^+ \rightarrow 10CO_2 + 2Mn^{+2} + 8H_2O$$

The reaction is rather slow at room temperature and so must be carried out at a somewhat elevated temperature. A small quantity of orthophosphoric acid is added to the titration mixture in order to complex the Fe^{+3} ion in a colorless form so that the end-point is more easily detected.

Part B. Analysis for Iron

In this determination fresh samples of the iron coordination compound are first treated with concentrated sulfuric acid. The concentrated sulfuric acid has a strong affinity for water and causes the decomposition of the oxalate ion present in the complex into CO and CO_2. The reaction is:

$$K_3[Fe(C_2O_4)_3] \bullet 3H_2O + 6H_2SO_4 \xrightarrow[H_2SO_4]{\text{conc}} 3K^+ + Fe^{+3} + 6HSO_4^- + 6H_2O + 3CO + 3CO_2$$

The resulting solution, which now contains Fe^{+3}, is diluted with water and treated with a sample of zinc which has been amalgamated with a small amount of mercury to increase its surface reactivity. The zinc quantitatively reduces the Fe^{+2} according to the reaction:

$$2Fe^{+3} + Zn \rightarrow 2Fe^{+2} + Zn^{+2}$$

The solution containing Fe^{+2} ion is then separated from the zinc amalgam by filtration. The reaction with permanganate is:

$$5Fe^{+2} + MnO_4^- + 8H^+ \rightarrow 5Fe^{+3} + Mn^{+2} + 4H_2O$$

Safety:

Oxalate: Poison! 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

Dilute hydrochloric acid (HCl): 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

Phosphoric Acid (H_3PO_4) Sulfuric acid (H_2SO_4): can harm eyes, skin, and clothing. Handle with care. Very hazardous in case of skin contact, of eye contact, of ingestion, of inhalation. Liquid mist may produce tissue damage on mucous membranes of eyes, mouth and respiratory tract. Skin contact may produce burns. Any acid spilled on the skin or splashed into your eyes must be rinsed with a large volume of water

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Potassium Permanganate: Hazardous in case of skin and eye contact, of ingestion, of inhalation. Slightly hazardous in case of skin contact. Possibly corrosive to eyes and skin. Inhalation of dust will produce irritation to gastro-intestinal or respiratory tract.

Mercury (II) chloride: Extremely hazardous in case of ingestion, of inhalation. Very hazardous in case of skin contact and of eye contact. Corrosive to eyes and skin. Eye contact can result in corneal damage or blindness. Skin contact can produce inflammation and blistering.

Part A. Analysis for Oxalate Ion

Procedure

On the analytical balance weigh out three samples (approximately 0.10g ± .1mg) of the iron coordination compound into individually labeled 250 ml. Erlenmeyer flasks. The following weighing procedure is to be carried out:

a. Tear a piece of weighing paper provided by the instructor.
b. Tap sufficient sample from the test tube onto the weighing paper until it is .10g ± .1mg. Weigh to .0001 g.
c. Transfer the sample to the labeled Erlenmeyer flask.
d. Using the same piece of weighing paper, repeat the procedure for the second and third samples.

To each flask add 6 ml. of 6M H_2SO_4, 2 ml. of conc. H_3PO_4 and dilute to about 50 ml. with deionized water. Obtain in a clean, dry beacker about 50 ml. of standardized solution of approximately molarity 0.015 M; rinse and fill your buret with this solution. Be sure to record the molarity of the standardized $KMnO_4$. Heat each of the samples in turn to just below the boiling point and titrate the hot solution immediately with the permangate solution.

*Note: Get your sample sizes as close of 0.10 g because if they are too large you will have to refill the buret in the middle of the titrations.

The end point of the titration occurs when the pink color of the last drop of permanganate added persists for at least 15 seconds.

Dispose of the titration mixture in the waste container provided.

From your data calculate for each sample the number of moles of $C_2O_4^{-2}$ ion in the sample, the mass of $C_2O_4^{-2}$ ion in the sample, and the percent by mass of $C_20_4^{-2}$ in the compound. Compare the average value for this last quantity with the percent by weight of $C_2O_4^{-2}$ calculated from the accepted formula of the compound.

Data:

Molarity of standardized $KMnO_4$ solution _____

SAMPLE NO.	1.	2.	3.
Mass of sample			
Final burette reading			
Initial burette reading			
Volume of $KMnO_4$			

Calculations: (Give setups for each of the samples.)

1. Moles of $C_2O_4^{-2}$ ion in sample
   sample 1
   sample 2
   sample 3

2. Mass of $C_2O_4^{-2}$ ion in sample
   sample 1
   etc…

3. Percent by mass of $C_2O_4^{-2}$ in the compound
   sample 1
   etc…

4. Average percent by mass of $C_2O_4^{-2}$ in the compound

5. Percent by mass of $C_2O_4^{-2}$ calculations from the accepted formula of the salt.

(Summarize the results of your calculations in the following table setup in <u>your notebook.</u>)

Calculations (Summary)

Sample	1	2	3
1. Moles of $C_2O_4^{-2}$ in sample			
2. Mass of $C_2O_4^{-2}$ in sample			
3. Percent by mass of $C_2O_4^{-2}$ in compound			
Average percent by mass $C_2O_4^{-2}$			
Percent by mass $C_2O_4^{-2}$ in compound calculated from formula			

Part B. Analysis for Iron

Procedure:

Using an analytical balance weigh out three samples (approx. 0.35g± .1mg each) of the iron coordination compound into individually labeled 250 mL Erlenmeyer flasks. Use the weighing procedure followed in the C_2O_4 part of the experiment.

THE NEXT OPERATION MUST BE DONE UNDER THE HOOD, since poisonous carbon monoxide is produced. To each of the samples carefully add 4mL of concentrated H_2SO_4 and heat each sample on the hotplate. When the last of your samples begins to dissolve, start timing and continue to heat for an additional twenty-five (25) minutes. Carefully remove your flasks from the hotplate and allow them to cool to room temperature. Keep the flasks under the hood. While the flasks are cooling, prepare the zinc amalgam as follows:

> Weigh out approximately 15 g of granulated zinc into a 125 mL Erlenmeyer flask and add approximately 30 mL of 1.5 % $HgCl_2$ in 1 M HCl. Shake the mixture for several minutes, pour off the excess solution, and wash the amalgam three times with 30 mL separate portions of deionized water.

Dispose of the solution in the waste container provided.

Write an equation for the reaction of Zn with $HgCl_2$.

After allowing the flasks to cool add 20 mL of deionized water to each sample: Add the water slowly and carefully, since a large amount of heat will be evolved and spattering of the concentrated acid may occur if you are not careful. ONLY AFTER ADDING THE WATER MAY THE FLASKS BE TAKEN OUT OF THE HOOD BACK TO YOUR BENCH.

Transfer the zinc amalgam to your watch glass and divide it into three portions, adding one portion to each of the three flasks. Swirl the flasks and then cover each flask with parafilm. Poke a hole in the parafilm and place your flasks in your drawer until the next laboratory period. You need a hole in the parafilm because some hydrogen gas will be produced and the flask should be able to vent itself.

When the reduction of the Fe^{3+} is complete, a Buchner funnel and three (3) filtering flasks from the side shelf and vacuum filter each of your samples into a separate filtering flask. Rinse the Erlenmeyer flask and zinc amalgam with three separate 10 mL portions of 2M H_2SO_4 collecting the solutions in the filtering flask along with the original filtrate in the filtering flask. Dispose of the used zinc amalgam and filter paper in the waste container provided.

Obtain about 50 ml of the standardized $KMnO_4$ in a clean and dry beaker. Rinse and fill the burette and titrate each of the samples in the filtering flasks to a faint pink end point. Dispose of the titration mixtures in the waste container provided.

From your data calculate for each sample the number of moles of Fe^{+3} in the sample, the mass of Fe^{+3} in the sample, the percentage by mass of iron in the compound. Compare the average value of this quantity with the percent by mass of iron calculated from the accepted formula of the compound. Finally from the results of parts A and B,

calculate the ratio of the number of moles of $C_2O_4^{-2}$ to moles of Fe in the compound and compare this with the theoretical ratio from the formula.

Set up the page in your notebook for the collection of data and calculation in the following way:

Data:

Molarity of $KMnO_4$ _____

Sample	1.	2.	3.
Mass of sample			
Final burette reading			
Initial burette reading			
Volume of $KMnO_4$ solution			

Calculations: (Give setups for each of the samples)

1. Moles of Fe^{+3} in sample
2. Mass of Fe^{+3} in sample
3. Percent by mass Fe in compound
4. Average percent by mass of Fe in compound
5. Percent by mass calculated from formula
6. Ratio of mole $C_2O_4^{-2}$ to moles Fe from experimental results (averages) (HINT: Calculate on basis of 100 g. sample.)
7. Theoretical ratio of moles $C_2O_4^{-2}$ to moles Fe

(Summarize the results of your calculations in the following table setup.)

Calculations (Summary)	1.	2.	3.
1. Moles of Fe^{+3} in sample			
2. Mass of Fe^{+3} in sample			
3. Percent by mass Fe in compound			
4. Average percent by mass Fe in compound			
5. Percent by mass calc. from formula			
6. Experimental mole $C_2O_4^{-2}$ / mole Fe			
7. Theoretical mole $C_2O_4^{-2}$ / mole Fe			

Questions:

1. Is it necessary to know the exact concentration of the $KMnO_4$ solution to determine the moles $C_2O_4^{-2}$ to moles Fe ratio for the compound? Explain.

2. Calculate the percent purity of your sample by comparing the experimental with the theoretical values for the percent Fe and for $C_2O_4^{-2}$ %.

   Use the following formula:

$$\% \text{ Purity} = 1 - \left(\frac{\text{Theoretical \% - Experimental \%}}{\text{Theoretical \%}} \right) \times 100$$

How do the two percentages compare? (In other words is the % Fe^{3+} purity close to the % $C_2O_4^{2-}$ purity) What are some possible explanations?

Summary: