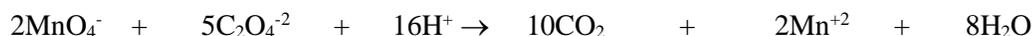


PART II: ANALYSIS OF IRON COORDINATION COMPOUND

In this experiment students will perform two independent analyses of the iron coordination compound synthesized in Part I. A redox titration with potassium permanganate solution will be utilized to determine both the iron and oxalate ion content of the $K_3[Fe(C_2O_4)_3] \cdot 3H_2O$ 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

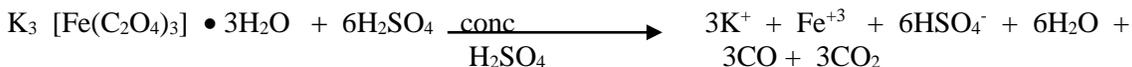
In this determination, the iron coordination compound is titrated directly with standard potassium permanganate in acid solution. The permanganate oxidizes the oxalate according to the equation:



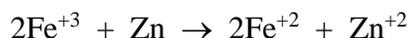
The reaction is rather slow at room temperature and so must be carried out at a somewhat elevated temperature. A small quantity of orthophosphoric acid is added to the titration mixture in order to complex the Fe^{+3} ion in a colorless form so that the end point is more easily detected.

Part B. Analysis for Iron

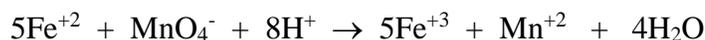
In this determination fresh samples of the iron coordination compound are first treated with concentrated sulfuric acid. The concentrated sulfuric acid has a strong affinity for water and causes the decomposition of the oxalate ion present in the complex into CO and CO_2 . The reaction is:



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:



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



Part A. Analysis for Oxalate Ion

Procedure

On the analytical balance weigh out three samples (approximately $0.10\text{g} \pm .1\text{mg}$) of the iron coordination compound into individually labeled 250 ml. Erlenmeyer flasks.

The weighing procedure:

- Tear a piece of weighing paper provided by the instructor.
- Tap sufficient sample from the test tube onto the weighing paper until it is $.10\text{g} \pm .1\text{mg}$. Weigh to $.0001\text{g}$.
- Transfer the sample to the labeled Erlenmeyer flask.
- 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 beaker about 50 ml. of standardized KMnO_4 solution of approximately molarity 0.015 M; rinse and fill your buret with this solution. Record the molarity of the standardized KMnO_4 . Heat each of the samples to just below the boiling point and titrate the hot solution immediately with the permanganate 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 titration.

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

DISPOSAL: Dispose of the titration mixture in the waste container labeled: “Solutions of MnO_4^- , Mn^{2+} , $\text{C}_2\text{O}_4^{2-}$, $\text{C}_2\text{O}_4^{2-}$, Fe^{3+} , and H_2SO_4 ”

From your data calculate for each sample the number of moles of $\text{C}_2\text{O}_4^{2-}$ ion in the sample, the mass of $\text{C}_2\text{O}_4^{2-}$ ion in the sample, and the percent by mass of $\text{C}_2\text{O}_4^{2-}$ in the compound. Compare the average value for this last quantity with the percent by weight of $\text{C}_2\text{O}_4^{2-}$ calculated from the accepted formula of the compound.

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

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:

1. Mass of $\text{C}_2\text{O}_4^{2-}$ ion in sample
sample 1
etc...
2. Percent by mass of $\text{C}_2\text{O}_4^{2-}$ in the compound
sample 1
etc...
3. Average percent by mass of $\text{C}_2\text{O}_4^{2-}$ in the compound
4. Percent by mass of $\text{C}_2\text{O}_4^{2-}$ calculations from the accepted formula
of the salt.

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

Calculation Summary Table

Sample	1	2	3
1. Mass of $\text{C}_2\text{O}_4^{2-}$ in sample			
2. Percent by mass of $\text{C}_2\text{O}_4^{2-}$ in compound			
3. Average percent by mass $\text{C}_2\text{O}_4^{2-}$			
4. Percent by mass $\text{C}_2\text{O}_4^{2-}$ in compound calculated from Chemical formula			

Part B. Analysis for Iron

Procedure:

Using an analytical balance weigh out three samples (approx. $0.35\text{g} \pm .1\text{mg}$ each) of the iron coordination compound into individually labeled 125 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 4 mL 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 about 15 g of granulated zinc into a 125 mL Erlenmeyer flask and add about 0.45 g of HgCl_2 and 30 mL of 1 M HCl . Shake the mixture for several minutes, pour off the excess solution, and wash the amalgam three times with 30 mL portions of deionized water. All solutions are disposed in the waste container provided.

Write an equation for the reaction of Zn with HgCl_2 .

DISPOSAL: Any excess HgCl_2 should be disposed in the container labeled “ 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 lightly stopper them with corks and place them in your drawer until the next laboratory period. You do not want to push the corks down tight 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, obtain a Buchner funnel and three (3) filtering flasks from the side shelf and vacuum filter each of your samples into separate filtering flasks. For each sample rinse the Erlenmeyer flask and zinc amalgam with **three** 10 mL portions of 2M H_2SO_4 collecting the solutions in the filtering flask along with the original filtrate in the filtering flask.

DISPOSAL: Dispose of the used zinc amalgam and filter paper in the waste container labeled “Amalgam solid”

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.

DISPOSAL: Dispose of the titration mixtures in the waste container labeled “Solutions of: MnO_4^- , Mn^{2+} , $\text{C}_2\text{O}_4^{2-}$, Fe^{3+} , and H_2SO_4 ”

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 $\text{C}_2\text{O}_4^{2-}$ 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 NO.	1.	2.	3.
Mass of sample			
Final burette reading			
Initial burette reading			
Volume of KMnO_4			

Calculations:

1. Mass of Fe^{+3} in sample
2. Percent by mass Fe in compound
3. Average percent by mass of Fe in compound
4. Percent by mass calculated from formula

5. Ratio of mole $\text{C}_2\text{O}_4^{2-}$ to moles Fe from experimental results (averages) (HINT: Calculate on basis of 100 g. sample using the % ave. mass Fe and % ave. mass $\text{C}_2\text{O}_4^{2-}$.)
6. Theoretical ratio of moles $\text{C}_2\text{O}_4^{2-}$ to moles Fe

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

Calculations (Summary)

	1.	2.	3.
1. Mass of Fe^{+3} in sample			
2. Percent by mass Fe in compound			
3. Average percent by mass Fe in compound			
4. Percent Fe by mass calc. from Chemical formula			
5. Experimental mole $\text{C}_2\text{O}_4^{2-}$ / mole Fe			
6. Theoretical mole $\text{C}_2\text{O}_4^{2-}$ / mole Fe			

Question

Calculate the percent purity of your sample by comparing the experimental with the theoretical values for the percent Fe and for $\text{C}_2\text{O}_4^{2-}$ %.

Use the following formula:

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

How do the two compare? What are some possible explanations?

Summary: