

Experiment 4A-C, Analysis of Oxalate Complex

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Abstract:

The goal of this experiment is to analyze the composition of an Iron(III) Oxalate complex ($K_w[Fe_x(C_2O_4)_y] \cdot zH_2O$) through the volumetric analysis oxidation-reduction titration and spectrophotometry. The experiment is divided into three parts. The first part involves the synthesis of the Iron(III) Oxalate Complex that will be analyzed by the consecutive parts. The mass of the Iron(III) Oxalate Complex obtained from a 1.2030 g $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ was 0.9623 g. In the second part analyzes the oxalate content in the complex. A portion of the Iron(III) Oxalate Complex is dissolved and titrated through a standardized solution of 0.0100 M of $KMnO_4$ to determine the weight percentage of the oxalate ligands in the Iron(III) Oxalate Complex, the result weight % of the oxalate was $53.2\% \pm 1.6\%$ and the number of mmoles of oxalate in a 100.0 g Iron(III) Oxalate Complex is $604 \text{ mmoles} \pm 18 \text{ mmoles}$. The third part of the experiment analyzes the iron content in the Iron(III) Oxalate Complex through spectrophotometry to find weight% of iron in the complex. The weight% of Iron(III) in the Iron(III) Oxalate Complex was $7.08\% \pm 0.92\%$ and the number of mmoles of iron in a 100.0 g Iron(III) Oxalate Complex is $127 \text{ mmoles} \pm 8 \text{ mmoles}$. Using the composition of Iron(III) and Oxalate, the compound was determined to be $K_7[Fe(C_2O_4)_5] \cdot 3H_2O$. Using the correct composition of the complex obtained from previous calculations, the mass of $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ and the mass of complex collected the percent yield was calculated to be 63.86%.

Experimental:**Experiment #4A (Synthesis of an Iron (III) oxalate complex):**

The experiment started with setting the filtering crucible to later collect the Potassium Iron(III) Oxalate Complex crystals by first drying in the in the oven and then cooling it in a desiccator. 1.2030 g g of $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ was dissolved in a clean 50-mL beaker with 3-mL of DI water containing 1-3 drops of 6M sulfuric acid. The solution was swirled and heated with a Bunsen burner until the salt was dissolved. Then 6-mL of 1M oxalic acid measured with a small graduated cylinder was added to the iron(II) ammonium sulfate solution and a yellow precipitate of $Fe(C_2O_4)$ was formed. The solution was tested with a pH paper that resulted a wine red color, meaning that the solution was a

strong acid and that it was suitable to continue (If the solution was not acidic enough, 6M H_2SO_4 would be added). The solution was heated with boiling stones in order to help dissolve the precipitates. The beaker with the solution was covered with a glass watch to avoid contamination, while cooling and waiting for the precipitate to settle. The clear solution was extracted leaving the precipitate in the beaker. The precipitate was washed 3 times with 4-5mL of hot DI water by swirling and waiting for the precipitate to settle. The supernatants were extracted and put together in waste beaker in hood.

3 mL of 2M KMnO_4 is added to the solution in a drop wise matter, resulting a solution with orange precipitate. The solution was warmed to 40.0 °C and 10 mL of 6% hydrogen peroxide solution was added to the warmed potassium iron(III) oxalate complex solution. The result, a brownish precipitate, was heated to the boiling point then 2mL of 1 M oxalic acid was added drop-wise and 15 more were added. The result was a clear yellow-green solution.

6 mL of 95% of ethanol was added to the yellow-green solution while scratching the inside wall with a stirring rod. Some crystals were formed and settled in the bottom of the solution. The solution was heated for the crystals to be dissolved, wrapped in aluminum and stored in the drawer overnight. Green crystals were formed in the bottom of the beaker the next day. The crucible was vacuumed and the crystals left in the crucible were air-dried for around $\frac{1}{2}$ and hour. The crucible with the crystal was weighed and the result product weighted 0.9623 g after deducting the initial crucible and the filter paper.

Results:

Experiment 4A (Synthesis of Iron(III) Oxalate Complex):

DATA:

Recorded mass of $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ used (Step #1):

First trial*: 1.2000 g

Second trial: 1.2030 g

Trial #1* was immediately aborted because of loss of significant amount of solution during an experimental error.

Recorded mass of green crystal product of Iron oxalate complex (Step #7):

	Crucible + filter paper	Crucible + Filter paper + Crystal	Crystal
Trial #1*	21.8472 g	Bumped, Sample aborted	none
Trial #2	21.8472 g	22.8095 g	0.9623 g

Trial #1* was immediately aborted because of experimental error with the “bumping”

OBSERVATION:

$\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ used reflected a light green/white color, its smell was similar to chlorine's. Two drops of sulfuric acid was used, and after adding the oxalic acid, the solution turned a very dark yellow color solution with tiny yellow precipitate). A pH test was performed with the pH paper, and the color displayed a wine red, which meant that the solution was very acidic and suitable to continue. It is worth mentioning that it was suspected some loss of iron oxalate in the process of transferring. After heating the yellow precipitate settled in the bottom of the beaker and the solution was very clear yellow. As far concerned, little to no precipitate seemed to be accidentally removed while washing, if so the error would be negligible. When potassium oxalate was added the precipitate turned orange and after adding peroxide the solution turned into a very light green color.

There were two trials for this part of the experiment because the first solution “bumped” when heating while adding hydrogen peroxide. Therefore the first trial with 1.2000g of $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ was disposed and another trial starting with 1.2030g of $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ was performed with the same procedure. The result after leaving the solution overnight was a green solution with tinny green crystals with around 5-millimeter radius. After air-drying, the crystals had a jasmine green color with some tiny white powder on top of the crystals, but not within.

No Q-test is needed because there was only one result.

Experiment 4B (Analysis of Oxalate in Iron (III) oxalate complex):

DATA:

Recorded Molarity of KMnO_4 labeled on bottle (Step #1): 0.0100 M

Recorded mass of Iron(III) Oxalate Complex (Step#2):

Sample #1	Sample #2	Sample #3	Sample #4
0.1015 g	0.1022 g	0.1017 g	0.1004 g

Recorded KMnO₄ volume (initial-final volume) (Step #5):

Sample	Initial	final	Total volume of KMnO ₄ used
#1	4.20 mL	28.55 mL	24.35 mL
#2	12.10 mL	36.40 mL	24.30 mL
#3	17.30 mL	42.35 mL	25.05 mL
#4	18.95 mL	43.25 mL	24.30 mL

Recorded temperature when heating solution containing potassium iron oxalate complex dissolved in acid (Step #5):

Sample #1	Sample #2	Sample #3	Sample #4
70.0 °C	68.0 °C	67.0 °C	65.0 °C

OBSERVATIONS:

Trial #1:

After dissolving the crystal with ~35mL of sulfuric acid-DI water solution, the result solution was a yellow color. As titration continued, the solution became clearer. The end point reached when solution displayed a very light and beautiful pink color. The volume was read on the top of the meniscus since the permanganate solution was too dark to read the bottom of the meniscus. However all of the readings were consistently read on the top of the meniscus.

Trial #2:

Similar to the first trial, the color of the solution was a clear yellow color after dissolving and heating. As titration continued, the solution also became clearer until it

reached the end point. The color of the end point was a very faint pink that was slightly darker than the pink in the first trial, however it is still considered "very ideal."

Trial #3:

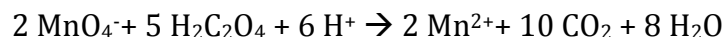
Similar to the previous trials, the color of the solution was initially a clear yellow, and as the titration continued the solution became clearer until the end point was reached. The color of the end point was a very faint pink that persisted for 30 seconds before it disappeared.

Trial #4:

Similar to the previous trials, the color of the analyte was initially a clear yellow and as the titrant was added the solution became clearer until the end point was reached. The color of the end point was a pink slightly darker than the previous ones, it is suspected to have overshot ½-1 drop.

CALCULATIONS:

The following calculations are based on the chemical reaction below, that determines the molar ratio between the limiting reagent and the reactant used.



All calculations are based on the following general formula:

1. To find the total mass of $\text{C}_2\text{O}_4^{2-}$ using the volume and the concentration of KMnO_4

$$[\text{KMnO}_4] \times (\text{volume, KMnO}_4) \times \frac{5 \text{ mol } \text{C}_2\text{O}_4^{2-}}{2 \text{ mol } \text{MnO}_4^-} \times (\text{molar mass, } \text{C}_2\text{O}_4^{2-}) = (\text{total mass of } \text{C}_2\text{O}_4^{2-})$$

Total mass of $\text{C}_2\text{O}_4^{2-}$

Trial #1:

Trial #2:

Trial #3:

Trial #4:

2. To determine the weight percent of $C_2O_4^{2-}$ using the total mass of $C_2O_4^{2-}$

$$\text{Weight Percent (\%)} = \frac{\text{mass of } C_2O_4^{2-}}{\text{mass of Iron(III) Oxalate Complex}} \times 100\%$$

Weight Percent of $C_2O_4^{2-}$

Trial #1:

Trial #2:

Trial #3:

Trial #4:

Q-test is not needed because there was no value that seemed to be out of place or any significant experimental error that might have jeopardized the data.

Calculation of the average percent weight of $C_2O_4^{2-}$:

Calculation of the standard deviation, σ :

Calculation of RSD%:

Average (% weight of $C_2O_4^{2-}$) $\pm 2\sigma$ =

3. Millimoles of $C_2O_4^{2-}$ that would be present in 100.0g complex. This information will be needed later in the lab in the "Experiment 4A-C "Wrap up Calculations":

$(\text{Weight percent of } C_2O_4^{2-}) \times (100.0\text{g of complex}) \div (\text{molar mass of } C_2O_4^{2-}) = (\text{moles of } C_2O_4^{2-})$

Millimoles of $C_2O_4^{2-}$ that would be present in 100.0g complex:

Trial #1:

Trial #2:

Trial #3:

Trial #4:

Q-test is not needed because there was no value that seemed to be out of place or any significant experimental error that might have jeopardized the data.

Calculation of the average millimoles of $C_2O_4^{2-}$:

Calculation of the standard deviation, σ :

Calculation of RSD%:

Average millimoles of $C_2O_4^{2-} \pm 2\sigma$ in 100.0 g complex:

Experiment 4C (Analysis of Iron in Iron (III) oxalate complex):

DATA:

Recorded mass of $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ (Part I Step #1): 0.12002g

Recorded mass of “**unknown” Iron(III) Oxalate Complex (Part II Step #1) : 0.1017 g

Recorded pH paper color for “unknown” solution (Part II Step#4): Orange (~pH=5, acidic)

Table 1

Recorded amount of DI Water added for dilution (Part III Step #2):

Sample	Initial value	Final value	Total DI water
#1 (known)	10.10 mL	16.10 mL	6.00 mL
#2(known)	28.10 mL	33.35 mL	5.25 mL
#3(known)	12.00 mL	16.50 mL	4.50 mL
#4(known)	21.00 mL	24.75 mL	3.75 mL
#5(known)	22.10 mL	25.10 mL	3.00 mL
#6 (unknown)	16.10 mL	22.10 mL	6.00 mL
#7(unknown)	33.35 mL	38.60 mL	5.25 mL
#8 (unknown)	16.50 mL	21.00 mL	4.50 mL
#9(unknown)	24.75 mL	28.50 mL	3.75 mL
#10 (unknown)	25.10 mL	28.10 mL	3.00 mL
extra *known	0.00 mL(grad cyl)	0.00 mL(grad cyl)	6.50 mL

Extra* was a new dilution for known sample in order for the transmittance for both unknown and known sample to overlap.

** Known solution refers to Iron(II) Bipyridyl Complex, unknown solution refers to the Iron(III) Oxalate Complex (in Bipyridyl sol)

Table 2.

Recorded volume (known and unknown) solution (Part III Step #2):

Sample	Initial value	Final value	Total solution used
#1 (known)	31.50 mL	33.00 mL	1.50 mL
#2 (known)	33.00 mL	35.25 mL	2.25 mL
#3 (known)	35.25 mL	38.25 mL	3.00 mL
#4 (known)	38.25 mL	42.00 mL	3.75 mL
#5 (known)	42.00 mL	46.50 mL	4.50 mL
#6(unknown)	32.00 mL	33.50 mL	1.50 mL
#7 (unknown)	36.80 mL	39.05 mL	2.25 mL
#8(unknown)	39.05 mL	42.05 mL	3.00 mL
#9(unknown)	42.05 mL	45.80 mL	3.75 mL
#10(unknown)	43.70 mL	48.20 mL	4.50 mL
Extra* known	Graduated cyl	Graduated cyl	1.00 mL

Table 3.

Table of DI water, solution and the T% measured from the spectrophotometer (Part III Step #7):

Sample	Total solution used	Total DI water	T%
#1 (known)	1.50 mL	6.00 mL	48.4%
#2(known)	2.25 mL	5.25 mL	33.4%
#3(known)	3.00 mL	4.50 mL	23.6%
#4(known)	3.75 mL	3.75 mL	16.2%
#5(known)	4.50 mL	3.00 mL	13.0%
#6 (unknown)	1.50 mL	6.00 mL	72.6%
#7(unknown)	2.25 mL	5.25 mL	59.0%
#8 (unknown)	3.00 mL	4.50 mL	53.4%
#9(unknown)	3.75 mL	3.75 mL	46.0%
#10 (unknown)	4.50 mL	3.00 mL	39.0%
extra *known	1.00 mL	6.50 mL	61.0%

Table 4*

Concentration and absorbance of known solution:

Sample	Concentration of solution	Absorbance
#1 (known)	2.44×10^{-5} M	0.315
#2(known)	3.66×10^{-5} M	0.476
#3(known)	4.88×10^{-5} M	0.627
#4(known)	6.10×10^{-5} M	0.790
#5(known)	7.32×10^{-5} M	0.886
extra *known	1.63×10^{-5} M	0.215

Data from Table 4* does not come from observation but from summarized calculations (3 and 4) in the “Calculation” section below. The table serves as a convenience for the graphing of Beer’s Law Plot on the Absorbance vs. Concentration of “known” solution.

OBSERVATION:

Part I and Part II:

The “known” ($\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$) solid displays a light green color. When Bipyridyl was added, the solution turned into a bright red color. Whereas the Iron(III) Oxalate Crystal has jasmine green, fairly small pieces with some green-white powder on top. The precipitate was harder to settle, it was centrifuged 3 times before it settled. The precipitate was white with a shade of green. It is noted that the supernatant was transferred to multiple centrifuge tubes when centrifuging, therefore there might be loss of liquid in between. The final solution for the “unknown” was a bit cloudy, light yellow green substance that turned into pink-red slightly brighter than the “known” solution after adding Bipyridyl.

Part III:

The instrument used for measuring the transmittance was a digital Spectrophotometer, therefore the transmittance level measured were fairly accurate from the machine. While measuring transmittance level of unknown, 3 of the unknown values

fell outside of the known range, therefore an extra* known was done with a new dilution in order to expand the range of the known and include the unknown values.

Calculation:

The following calculations are based on the concept below:

The purpose of this experiment is to find the weight % of iron in complex by finding the concentration of the initial standard solution of Iron(III) Oxalate Complex. The absorbance and the concentration of the “known” solution will serve as a range to help find the concentration of “unknown.”

1. Concentration of the iron(II) in standard solution:

$$[Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O] = \frac{\text{mass of } Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O \text{ (g)}}{\text{molar mass of } Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O \text{ } (\frac{g}{mol})} \times \frac{1}{\text{Volume (L)}}$$

Concentration of the iron(II) in standard solution:

2. Final concentration of iron(II)/ bipyridyl complex after dilution:

Final Concentration= (Initial Concentration) (Initial Volume/ Final Volume)

$$C_f = C_0 \times \frac{V_0}{V_f}$$

[Conc. of diluted Iron(II) bipyridyl complex] =

[conc. of standard sol] (volume of standard solution added / total volume)

$$[\text{Conc. of diluted Iron(II) bipyridyl complex}] = [\text{conc. of standard sol}] \left(\frac{2.00 \text{ mL}}{50.00 \text{ mL}} \right)$$

Final concentration of "Known" Solution:

3. To find the final concentration of each perspective "known" sample after dilution

Final Concentration= (Initial Concentration) (Initial Volume/ Final Volume)

$$C_f = C_0 \times \frac{V_0}{V_f}$$

$$["\textit{known"} \textit{ sample}] = [\textit{standard "known"} \textit{ sol'n}] \times \frac{(\textit{volume sol'n added})}{(\textit{total volume, 7.50 mL})}$$

The concentration of unknown sample cannot be yet known since the molar mass of unknown is still unknown and the initial concentration cannot be yet calculated.

The initial concentration of the known sample is derived from the result of the final concentration of Iron(II) Bipyridyl complex solution from Part II: (0.000122 M of "known" solution)

Sample #1 (known):

Sample #2 (known):

Sample #3 (known):

Sample #4 (known):

Sample #5 (known):

Extra* (known solution):

4. The absorbance is needed to plot the Beer's Law Plot of "known" solution, in order to find the concentration of Iron(III) Oxalate Complex in each sample (#6-#10)

Therefore the transmittance measured is converted to absorbance:

$$A = \log \frac{100\%}{\%T}$$

Sample #1 (known):

Sample #2 (known):

Sample #3 (known):

Sample #4 (known):

Sample #5 (known):

Sample #6 (unknown):

Sample #7 (unknown):

Sample #8 (unknown):

Sample #9 (unknown):

Sample #10 (unknown):

Extra* (known solution):

The results of absorbance and concentration of “known” solutions (Sample #1-5 and extra* known) from calculation 3 and 4 are summarized in Table 4, “Data” section to facilitate the graphing of Beer’s Law Plot (attached page) and find the linear regression equation.

The resulting linear regression equation: $y = 12117x + 0.0258$

5. The absorbance equation $A = \epsilon bc$ is used in order to find the concentration of each perspective “unknown” solutions (Sample #6-#10).

The slope of the linear regression obtained from Beer’s Law graph from known solutions is equal to ϵb therefore using absorbance of “unknown solution,” the concentration from each perspective solution can be found.

Linear regression line from Beer’s Law graph (page attached): $y = 12117x + 0.0258$

where x = concentration and y = absorbance.

Sample #6 (unknown):

Sample #7 (unknown):

Sample #8 (unknown):

Sample #9 (unknown):

Sample #10 (unknown):

6. 1 To find the initial concentration of unknown iron(III) oxalate complex(with bipyridyl) before samples were diluted and the concentration of the standard solution we must use the concept below:

Since Fe: Bipyridyl= 1:1 therefore stoichiometry should not be concerned.

Final Concentration= (Initial Concentration) (Initial Volume/ Final Volume)

$$C_f = C_0 \times \frac{V_0}{V_f}$$

except C_0 will be the targeting value. Therefore $C_0 = C_f \times \frac{V_f}{V_0}$

[unknown diluted in bipyridyl sol'n]

$$= [\text{diluted samples}] \times \frac{(\text{total volume, 7.50 mL})}{(\text{volume of sol'n added})}$$

[Standard "unknown"]

$$= [\text{"unknown" diluted in bipyridyl sol'n}] \times \frac{(\text{vl unknown} - \text{bipy})}{(\text{vl standard "unk"})}$$

[Standard "unknown"]

$$= [\textit{diluted samples}] \times \frac{(\textit{total volume, 7.50 mL})}{(\textit{volume of sol'n added})} \times \frac{50.00 \text{ mL}}{2.00 \text{ mL}}$$

Concentration of 100mL Iron(III) Oxalate Complex solution:

Sample #6 (unknown):

Sample #7 (unknown):

Sample #8 (unknown):

Sample #9 (unknown):

Sample #10 (unknown):

Q-Test: Because suspect a data from Sample #6 (0.00117M) to be a possible outlier.

(datas: 0.00117 M, 0.00140 M, 0.00127 M, 0.00129 M, 0.00132 M)

$$\left| \frac{0.00117 - 0.00127}{0.00140 - 0.00117} \right| = 0.43$$

The Q_c value for five values is 0.64. Because $0.43 < 0.64$, therefore the 0.00117 M cannot be rejected, however the values are spread in wide range. Matters of precision will be later discussed.

Average Concentration of unknown iron(III) oxalate complex in 100mL:

Average concentration unknown iron(III) oxalate complex solution (mmol/mL) =

7. After finding the concentration of the Iron(III) Oxalate in 100.00 mL, the numbers of moles and mass of the Iron(III) can be found to calculate the iron weight %

To determine the millimoles of iron in the 100.00mL solution from the concentration of standard "unknown" solution:

Because the moles of the Iron(III) Oxalate Complex = moles of Iron (III) Therefore:

[Concentration of Iron(III) Oxalate Complex] (100.00 mL) = (millimoles of Iron(III))

Millimoles of Iron (III):

Sample #6 (unknown):

Sample #7 (unknown):

Sample #8 (unknown):

Sample #9 (unknown):

Sample #10 (unknown):

Average millimoles of iron(III):

Standard deviation of millimoles of iron(III):

RSD%:

Average millimoles of Iron(III) $\pm 2\sigma$:

8. To find the weight percent of Iron(III), first find the mass of Iron (III) by multiplying the molar mass of Iron (III) with the calculated millimoles of Iron(III):

$$(\text{moles of Iron(III)}) (\text{mm of Iron(III)}) = \text{mass of Iron(III)}$$

Then, using the mass of the Iron (III), find the weight percent:

$$\text{Weight Percent (\%)} = \frac{\text{mass of Iron(III)}}{\text{mass of Iron(III) Oxalate Complex}} \times 100\%$$

Therefore:

$$\text{Weight Percent (\%)} = \frac{(\text{moles of Iron(III)}) (\text{mm of Iron(III)})}{\text{mass of Iron(III) Oxalate Complex}} \times 100\%$$

The mass of the Iron(III) Complex is recorded from Part II Step #1.

Weight percent of Iron(III):

Sample #6 (unknown):

Sample #7 (unknown):

Sample #8 (unknown):

Sample #9 (unknown):

Sample #10 (unknown):

Average weight% of Iron(III):

Standard deviation of weight% of Iron(III), σ :

RSD%:

Average weight% of Iron(III) $\pm 2\sigma$:

9. To determine the number of millimoles of iron that would be present in 100.0 g complex. This information will be used for the "Experiment 4A-C "Wrap up Calculations.")

$(\text{Weight percent of Iron(III)}) \times (100.0\text{g of complex}) \div (\text{molar mass of Iron(III)}) = (\text{moles of Iron(III)})$

Millimoles of Iron(III) that would be present in 100.0g complex:

Sample #6 (unknown):

Sample #7 (unknown):

Sample #8 (unknown):

Sample #9 (unknown):

Sample #10 (unknown):

Calculation of the average millimoles of Iron(III):

Calculation of the standard deviation, σ :

RSD%:

Average millimoles of Iron(III) $\pm 2\sigma$ =

Experiment 4A-C (Wrap up Calculations):

The purpose of this section of calculations is to find the composition of the Iron(III) Oxalate Complex: $K_w[Fe_x(C_2O_4)_y] \cdot zH_2O$. The values w, x, y, z . Will be found through the molar ratio between the composites. At last, the percentage yield will be confirmed.

A. To determine x and y values, the ratio of oxalate to iron ratio must be found. The data will be drawn from the number of millimoles of oxalate in 100.0g Iron(III) Oxalate Complex calculated in Experiment #4B Calculation 3 and the number of millimoles of Iron(III) in 100.0g Iron(III) Oxalate Complex calculated in Experiment #4C Calculation 9.

Millimoles of Oxalate in 100.0g Complex: Millimoles of Iron(III) in 100.0g Complex = $y : x$

Ratio of Oxalate to Iron(III):

B. To determine the w value of potassium, x, y values will be used to balance the compound to an electrically neutral compound using the charges of each perspective ion.

Fe: +3

K: +1

C_2O_4 : -2

x :

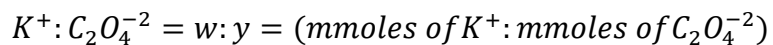
y :

Number of potassium ions:

C. To help find the z value for composition of water, it is needed to know the millimoles and weight percentage of potassium ion.

C-1-The millimoles of potassium ion in the Iron(III) Oxalate Complex will be found from the ratio of potassium ion to both Iron(III) and Oxalate and the millimoles of each perspective composite in 100.0g of Iron(III) Oxalate Complex.

$$K^+ : Fe^{+3} = w : x = (\text{mmoles of } K^+ : \text{mmoles of } Fe^{+3})$$



Millimoles of Potassium ion according to ratio $K^+ : Fe^{+3}$

Millimoles of Potassium ion according to ratio $K^+ : C_2O_4^{-2}$:

C-2. To determine the weight percent of K^+ in 100.0 g Iron(III) Oxalate Complex using the total mass of K^+ :

$$(\text{moles of } K^+ \text{ in } 100.0\text{g}) (\text{mm of } K^+) = \text{total mass of } K^+$$

$$\text{Weight Percent (\%)} = \frac{\text{mass of } K^+}{\text{mass of Iron(III) Oxalate Complex (100.0 g)}} \times 100\%$$

Weight percent of K^+ in 100.0g Iron Oxalate Complex:

D. To determine the z value for composition of water, find the number of millimoles of water.

D-1. First, subtract weight in 100.0g Iron Complex of three species (Iron(III), Oxalate and Potassium ion) to 100.0g of Iron Complex. The remaining weight will be the weight of water.

(Total mass of Iron(III) Oxalate Complex (100.0g)) - (weight of Iron(III) g+ weight of Oxalate g+ weight of Potassium g)= (weight of water g)

Weight of water in Iron(III) Oxalate Complex:

D-2. Then using the mass of water in 100.0g complex, find the mmoles of water and compare to find ratio of $x:z$ using *mmoles of Fe^{+3} : mmoles of H_2O*

$$(\text{total mass of } H_2O) \div (\text{molar mass of } H_2O) = (\text{moles of } H_2O)$$

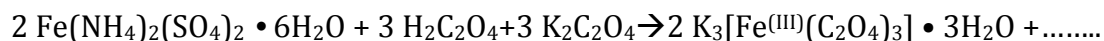
$$\text{mmoles of } Fe^{+3} : \text{mmoles of } H_2O = x : z$$

Number of H_2O (z):

The Empirical Formula calculated: $K_7[Fe(C_2O_4)_5] \cdot 3H_2O$

The true Empirical Formula (from Postlab of Experiment 4B #2f): $K_3[Fe(C_2O_4)_3] \cdot 3H_2O$

To determine the yield percent of the reaction the molar ration between the reactant and the product in the balanced equation must be considered: (The equation was drawn from Postlab of Experiment #4B, because the empirical formula calculated does not match):



Using the moles of $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ using its initial mass to find the theoretical mass of product through the molar ratio of $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ to $\text{K}_3[\text{Fe}^{(\text{III})}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}$ and find percent yield.

$$(\text{mass of } \text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}) \div (\text{mm of } \text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}) = (\text{moles of } \text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O})$$

$$(\text{moles of } \text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}) \times \frac{\text{moles } \text{K}_3[\text{Fe}^{(\text{III})}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}}{\text{moles } \text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}} \times (\text{mm of}$$

$$\text{K}_3[\text{Fe}^{(\text{III})}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}) = (\text{mass of } \text{K}_3[\text{Fe}^{(\text{III})}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O})$$

The mass of $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ is obtained from Experiment 4A Step#1 = 1.2030 g

Theoretical mass of $\text{K}_3[\text{Fe}^{(\text{III})}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}$:

The actual mass of $\text{K}_3[\text{Fe}^{(\text{III})}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}$ is recorded from Experiment #4A Step #7 = 0.9623 g

Percent yield:

$$\frac{\text{actual yield}}{\text{theoretical yield}} \times 100\%$$

The percent yield of $\text{K}_3[\text{Fe}^{(\text{III})}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}$:

Discussion:

The analysis of the synthesized Iron(III) Oxalate complex from Experiment #4A resulted the average weight percentages of $53.2\% \pm 1.6\%$ of oxalate from Experiment #4B and $7.08\% \pm 0.92\%$ of iron(III) content from Experiment #4C. From these weight percentages, the number of millimoles of both oxalate and iron(III) that would be in a 100.0 g complex, $604 \text{ mmol} \pm 18 \text{ mmol}$ and $127 \text{ mmol} \pm 8 \text{ mmol}$ respectively, were calculated in order to help determine the coordination number of the compound. The results from the calculations were: weight percent of 33.1% and 846-889 mmoles of potassium ion in 100.0 g of complex and 6.62% and 367 mmoles of water in 100.0 g of complex. The resulting calculated coordination compound was: $K_7[Fe(C_2O_4)_5] \cdot 3H_2O$. However, the calculated empirical formula did not correspond to the expected empirical formula, $K_3[Fe(C_2O_4)_3] \cdot 3H_2O$, therefore the percent yield was calculated using the expected empirical formula. The resulting percent yield from Experiment #4A was 63.86%.

The yield was calculated using the correct empirical formula, instead of the incorrect empirical formula calculated from the results. A yield of 63.86% is considered fair for the effectiveness on the synthesis of Iron(III) Oxalate Complex in Experiment #4A. There are several reasons for the synthesis to have a lower yield: First, as stated in the observation from Experiment #4A, it was suspected some loss of the Iron(II) ammonium sulfate hydrated salt in the process of transferring. Also, the possible loss of precipitate when washing or left in the stirring rod and glassware can attribute a smaller percent yield. An observation worth mentioning was the appearance of the crystals synthesized. The crystals were jasmine green small "rocks" with white powder on top. The white powder could be an indication of a source of error. The solution might not have been mixed well during the synthesis, resulting a possible product other than the one expected. This could have greatly affected the results of the analysis of the Iron(III) Oxalate Complex, and it might have attributed to the incorrect composition number in the empirical formula of the compound.

The calculated empirical formula $K_7[Fe(C_2O_4)_5] \cdot 3H_2O$ was way off the correct empirical formula, $K_3[Fe(C_2O_4)_3] \cdot 3H_2O$. As seen, the number of water compound is correct, the ratio of potassium to oxalate is slightly off, but the biggest difference is the ratio between iron(III) and oxalate. The calculated ratio of iron(III) and oxalate from the number

of millimoles of each respective iron ion and oxalate ligands, $127 \text{ mmol} \pm 8 \text{ mmol}$ and $604 \text{ mmol} \pm 18 \text{ mmol}$, was $1 : 4.76$, and according to Dalton's Law, the combination ratio must be whole numbers, therefore the ratio was rounded up to $1 : 5$. It is suspected that the main source of error laid on the iron(III) analysis in Experiment #4C rather than the oxalate analysis in Experiment #4B. This is also the reason why the weight percent of the potassium was calculated from theoretical number of mmoles of potassium ion in 100.0 g of complex, 846 mmoles , according to the number of mmoles of the oxalate instead of the iron's.

There are several reasons why the analysis iron(III) is suspected to be the main source of error. First, the standard deviation (2σ) weight % of iron(III), 0.92% , is considered relatively big compared to the average, 7.08% . This can be proven from the $\text{RSD}\% = 6.5\%$, meaning that the data has lower precision. Although a Q-test was performed with the calculated concentrations of the "standard unknown" solution with a possible outlier (Sample #6), the result was $0.43 (< 0.64, Q_c \text{ for } 5 \text{ values})$, therefore the data could not be rejected. However it was observed that the data had wider range, hence the bigger standard deviation.

Although low precision does not necessarily imply low accuracy, the fact that the experimental procedure for the analysis of iron (III) required more steps, exposes the results for much more room of error. For instance, as noted in the observations in Experiment #4C, the precipitate, calcium oxalate, from the "unknown" Iron (III) Oxalate complex solution, after adding calcium chloride solution, was harder to settle. The solution was centrifuged 3 times before continuing to the dilution with bipyridyl solution, and the supernatant used was still a bit "cloudy." Although the substance turned to the expected bright red color after adding bipyridyl solution, the un-extracted precipitate might have possibly reacted with the bipyridyl or interfered with the measurement of the transmittance during spectrophotometry. The accurate dilution measurement technique is also crucial, having an accurate measurement of the perspective samples of solution and water is important for the calculation of concentration of each sample. An off concentration can result inconsistency of the data. Also the handling of the cuvette is extremely important for the reading during spectrophotometry; fingerprints, water, or minor scratches can result off reading of the actual transmittance level. At last, the possibility for contamination

should be always considered in any experiment. In the case of the analysis of Iron (III) in Experiment #4C, contamination can cause reaction with the solution, or inaccurate readings of transmittance during spectrophotometry that might have possibly attributed to the low precision and the inaccuracy in the analysis of iron (III). Contaminants and particles in solution might have led the solution to have lower absorbance level, causing the underestimation of the weigh% of iron.

The results from the oxalate analysis from Experiment #4B seem to be far more reliable from the results from the analysis of iron(III). Looking at the results of the calculations for the weight% of oxalate drawn from the oxidation-reduction titration, there was no apparent outlier in the data, there was also no apparent experimental error that might have jeopardized the results, and therefore no Q-test was performed. The standard deviation (2σ), 0.8% is considered fairly small compared to the average weight% of oxalate, 53.2%. This can be proven from the RSD%=1.5%, which is considered “fairly precise.”

Although precision does not imply accuracy, it does mean that the data of the experiment is fairly consistent. Possible errors that might deviate from accuracy should be still considered. First, the possible loss of solid Iron(III) Oxalate Complex when transferred can create a difference between the expected dissolved mass and the actual dissolved mass, which can overestimate the weight % of the oxalate calculated and affect the later calculations for the composition of the compound. Another possible error is the variation of the color end point in the titration trials. Different ratios between the titrant and the analyte can create solutions that might have overshoot. For instance, as mentioned in the observations in Experiment #4B, the end point from all 4 trials vary from having a faint pink that disappeared in 30 seconds to a slight darker pink that is suspected to have overshoot $\frac{1}{2}$ to 1 drop of titrant. These can cause deviation during calculations for weigh% for oxalate from the actual value. Although, during volumetric analysis, splashes of titrant left on the flask walls are usually a potential error to consider, in Experiment #4B, however, the titrant is dark enough to notice any drops, therefore the potential error is minimal to negligible. At last, all reagents and solutions are exposed to contamination that might have affected the results.

In Experiment #4B, the volumetric analysis has proven to be a fairly accurate and reliable method for analytic chemistry. In this case, reduction-oxidation titration was used to help determine the weight % of a ligand in a compound. In real life, industries use redox titrations to determine the content in their product. For instance, food industries such as Heinz, uses redox titration to determine the acid content in their pickle liquor. In Experiment #4C, spectrophotometry has been proven to be quicker and more convenient when multiple samples need to be analyzed. In this case, samples with multiple concentrations helped analyzed the weigh% of iron. Although there is a much bigger room for error since there are several experimental procedures, spectrometry can be very consistent and fairly accurate if solutions are handled carefully. Spectrophotometry is widely used across the clinical and biology field. For instance, it is used to measure iron, metabolites and other body fluids in blood for clinical purposes.

Experiment #4A-C shows a fairly accurate determination of the empirical formula through the results of a wide variety of analysis suitable for different composites in compound, Iron(III) Oxalate Complex. While some composites are suited for titrations, others are suited for spectrophotometry, demonstrating a wide amount of options to perform analytical chemistry. In the real world, analytical chemistry is often used to analyze the composition of the atmosphere in factories and even to monitor the level of toxic substances in factory workers to ensure their health.