**Experiment 4 Writeup**

**Required Content**

**Excel Spreadsheet: Upload on Ted under “Expt 4 spreadsheet” (10 points)**

**Title: Synthesis and Analysis of an Iron(III)-Oxalate Complex**

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**Abstract (30 points for accuracy + 10 points general)**:

The final goal of the experiment was to create a Iron(III)-Oxalate Complex and to find its empirical formula. This experiment utilized methods of synthesis, reduction-oxidation titrations, and chemical absorbance to build the complex and then complete the empirical formula. Oxalic Acid was combined with Iron(II) ions and Hydrogen Peroxide to synthesize the Iron(III) crystals in the first portion of the experiment. The second portion utilized reduction of the Permanganate Ion and oxidation of the Oxalate ion to determine the mass percentage of the Oxalate ion in the sample through titrations. A known mass of the complex was first dissolved in Deionized water and Sulfuric Acid. a Potassium Permanganate solution of known concentration was then added in small volumes via titration to accurately find the moles of Permanganate Ions required to completely oxidize the Carbon atoms in the Oxalate. Based on the number of moles of Permanganate Ion added to the solution, a calculation was made that determined the mass percentage of Oxalate Ion in the sample. In the Third portion of the Experiment, the sample was diluted several times in parallel with a standard of known composition. The Iron(III) in the sample was first oxidized to Iron(II) to match the Iron(II) in the standard solution. Bipyridyl solution was added to both samples to create a red solution. The solution was then diluted several times to create a known calibration curve, which was then used to back calculate the initial concentration of the original Iron(II) Bipyridyl solution concentration, which was then used to calculate the number of moles of iron present, and given the number of moles present and the initial known mass of the complex added to the solution, the mass percentage could then be calculated. Based on the calculations, the empirical formula of the complex is as follows: K3[Fe(C2O4)3] · 5H2O. The complex of simply Fe(C2O4)3 does not have a balanced charge, so it is synthesized with extra potassium ions to balance out the charge. This solution was also once aqueous, so any leftover mass percentage after all of the calculations are assumed to be water molecules locked in crystal hydrates in the synthesized product. It is also required to know that this is an empirical formula of the compound, meaning that the formula is only in its lowest number state, and the actual composition of the complex may be some multiple of this empirical formula’s numbers of ions that comprise the complex.

**Introduction (20 points), < 3 pages (< 1500 words)**

* Provide brief background on the synthesis and coordination complexes, etc. You may utilize primary resources as needed.
* Provide background on techniques such as titration, redox titrations, and spectrophotometry (Beer's Law).
* Briefly discuss how the above information allows you to make and analyze the iron oxalate complex.

The use of synthesis is needed for the creation of new chemicals and new materials as well as the manufacture of already known materials. Synthesis in the case of this lab, utilizes the creation of coordination compounds, which are “the product of a Lewis acid-base reaction in which neutral molecules or anions (called ligands) bond to a central metal atom (or ion) by coordinate covalent bonds” (Purdue). The creation of these covalent bonds often causes the compound to fall out of solution and precipitate out, as observed in Experiment 1 with the Aluminum Hydroxyquinoline falling out of solution as the Ions bond together and create coordinate covalent bonds. The use of coordination complexes is present in many places, from photography to hemoglobin. A coordination complex basically entails having a metal ion, typically a transition metal, bonding with different ligands to create different compounds.

Techniques that will be used in this lab include titration setups, in which the burets will be used to accurately measure the volume of liquid of a known concentration into a flask containing a solution of known species and unknown concentration. The known concentration, volume, and species of the solution within the buret will allow later calculation of the quantitative contents of the flask. Generally, Titrations are used for Acid/Base volumetric analysis purposes, but they are also useful for redox titrations, as they allow the precise and consistent addition of a reducing agent (KMnO4 in this case) into an oxidizing solution. This experiment employs the use of redox reactions in the form of the Permanganate Ion reducing Oxalate ion to analyze the moles of oxalate present in a given sample. The difference with redox titrations and regular acid/base titrations is that redox titrations are not buffered in the sense that there is a massive amount of known titrant that needs to be added to the unknown to complete the titration. Redox titrations generally rely on the use of a catalyst to make the reaction go faster and more smoothly, allowing quick addition of titrant to the solution. The knowledge of how to add small half drops and small quarter drops is vital to not overshooting the end point of the titration, ensuring that a more accurate result will come out.

Spectrophotometry is used to measure the concentration of a certain species within a solution via measuring the absorption of light as it passes through the solution. In the case of this Experiment, Spectrophotometry was used to determine the concentration of Iron in a dilute solution of Iron(II) Bipyridyl. Spectrophotometry takes advantage of Beer’s Law, in which an Absorbance given from a Spectrophotometer can be used to calculate the concentration based on the known species’ absorption constant, ϵ, and the path length through the solution, b. In this experiment, a solution of known concentration will be made into known dilutions and be put into the spectrophotometer to make a calibration curve. This calibration curve will give the slope of the line, which will provide the value of ϵb, which will not change because epsilon of a species is a constant, and in this case, since the same cuvette will be used for every single trial, the value of b will be unchanged, leaving the value of C as an independent variable. With the knowledge of the calibration curve, a sample of unknown solution can be tested for absorbance and the absorbance can then be back compared to the calibration curve to find the concentration, since the relationship between concentration and absorbance is linear in this case.

With the knowledge of how much reducing agent needs to be added to a solution to balance the equation, the number of moles of target molecule, Oxalate in this case, can be back calculated to find the mass of the total oxalate in a sample, and with the mass of the oxalate, it is possible to calculate the mass percentage of the Oxalate with respect to the total mass of the initial product sample added to the solution. In Experiment 4C, the use of a calibration curve and back calculation of the concentration of iron atoms in the sample will allow the calculation of the moles of iron product added to the original flask before the first dilution. The calculation of the number of moles in that sample will give the mass of the iron with respect to the mass of the complex, these two numbers will allow the calculation of the mass percentage of the Iron in comparison with the rest of the compound. Use any present alkali ions (Potassium in this case) from the synthesis step to back fill any charge differences to balance the charge. With the knowledge of how many iron, potassium, and Oxalate ions needed to form the complex, any leftover mass percentage is assigned to water, as it fills in the hydrate between the gaps in the crystal lattice.

**Experimental (20 points), < 4 pages (<2000 words)**

* \*note that this procedure was used to make a double sample of crystals

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

A crucible was placed into an oven to dry for one hour, the dried crucible was then placed into a desiccator to cool. 2.3990 g of Iron(II) Ammonium Sulfate crystal was weighed and added to a clean 50-ml beaker. 6.01 ml of Deionized Water was added along with 6 drops of Sulfuric Acid to the beaker. The contents of the beaker were stirred until all the crystals had dissolved. 12.03 ml of 6M Oxalic Acid was added to the solution and the solution was thoroughly stirred. The pH was then checked with the use of pH paper. No additional sulfuric Acid was added to make the solution more acidic. A water bath was prepared using a 250-ml beaker and 10 boiling chips in which the 50 -ml beaker with the solution was immersed. The solution was allowed to be boiled for 5 minutes. The solution was constantly stirred in a gentle manner to prevent bumping in the beaker. The precipitate that was formed was then separated from the supernatant through decanting and washing with warm Deionized water that was separately prepared on the side of the hotplate in another part of the Lab. The supernatant and wash fluid was disposed in a designated waste container under the fume hood. The precipitate was washed a total of three times. 7.0 ml of 1.7 M Potassium Oxalate solution was added to the beaker with the precipitate. The solution was then stirred to dissolve the precipitate. After, dissolution, the solution was heated to 40° C and 20 ml of 6% Hydrogen Peroxide was added dropwise to the solution to oxidize the Iron(II) in the solution to Iron(III). The addition of Hydrogen Peroxide was done slowly to maintain the temperature at ~40°C to prevent the solution form bumping or boiling, as the oxidation reaction here is exothermic. After addition of the Hydrogen Peroxide, the solution was then heated to its boiling point and then immediately removed from the hot plate. 2 ml of 1M Oxalic Acid was added drop wise with constant stirring to the solution. 15 more drops of 1M Oxalic Acid was added to ensure that Oxalate Ion was not the limiting Reagent. The solution was allowed to cool to room temperature after covering the beaker with Parafilm. 10 ml of 95% ethanol solution was added to the beaker. The sides of the beaker were scratched with a stirring rod to encourage crystallization. The Solution was then reheated and stirred until all of the crystals had dissolved. The solution was then covered with Parafilm and the entire beaker was wrapped in Aluminum Foil to allow the formation of crystals over the course of 45 hours. The previously dried crucible was weighed and a vacuum filtration system was set up to separate the crystals form the supernatant. When filtering, the crucible was first washed in 95% ethanol. After filtration, the crystals in the crucible were partially covered with a paper towel to prevent degradation of the product (it is light sensitive). The sample was allowed to dry for 30 minutes before it was weighed to find the mass of the crystals. The mass of the crystals yielded in the synthesis step was 2.9009 grams.

Experiment 4B: Analysis of Oxalate Ligands in an Iron(III)-Oxalate Complex

The concentration of the Potassium Permanganate Solution was recorded. 4 samples of about 0.08 g Iron(III)-Oxalate crystals (henceforth referred to as “product”) were prepared the masses of each sample were carefully noted and were recorded. Each sample was then qualitatively transferred to a different Erlenmeyer flask. The labels and which sample went into each flask were then noted. To each flask, ~30 ml of Deionized Water and ~2 ml of 6M Sulfuric Acid was added, and each flask was then swirled to dissolve the crystals. ~ 80 ml of was obtained in a clean 150 ml beaker. A buret was set up and cleaned first with Deionized Water. ~5 ml of the Potassium Permanganate Solution was added to the buret and then the buret was closed using the stopcock and a finger. The buret was inverted several times so that the Potassium Permanganate solution would cover the entire surface. Fill the buret with ~40 ml of the Potassium Permanganate solution. Over a hot plate, heat the dissolved product until it reaches ~60° C. Record the starting volume of the Potassium Permanganate solution in the buret Slowly titrate the sample until it turns a pale pink. If the sample turns a shade of brown and does not turn back to pink or yellow, then the sample should be recreated and should be titrated again to obtain an actual value for the volume of the Potassium Permanganate. When the pink color persists for at least 30 seconds, the titration is finished. Record the ending volume of the titrations. Properly dispose of the titration products in the designated containers.

Experiment 4C: Analysis of Iron in an Iron(III)-Oxalate Complex

0.1269 g Iron(II) Ammonium Sulfate crystals were qualitatively added to a 100 ml volumetric flask. ~ 15 ml of Deionized Water and 0.53 mg of Ascorbic Acid were added to a separate 50 ml volumetric flask. A 2 ml volumetric flask was then used to pipette 2.0 ml of the 100 ml solution into the 50 ml volumetric flask. 15.0 ml of Bipyridyl solution was measured first in a graduated cylinder and then added to the 50 ml volumetric flask with the use of a funnel. Fill the graduated cylinder to the calibration line with the provided 4.7 pH buffer. Transfer the contents of the 50 ml volumetric flask into a 125 ml storage container. This solution will be the standard used later. the bottle was labeled as such. The volumetric flasks and graduated cylinders were then thoroughly cleaned with Deionized Water. 0.1132 g of product was measured out and quantitatively transferred into a 50 ml centrifuge tube with ~20 ml of Deionized Water. 5.1 ml of 9M Sulfuric Acid was added to the tube along with 15.03 ml of 0.5M Calcium Chloride solution. The tube was capped and allowed to sit for 5 minutes for a precipitate to form. The tube was then centrifuged and the supernatant was transferred into a 100 ml volumetric flask using a medicine dropper to decant the liquid and prevent precipitate from entering the flask. The flask was then filled to the calibration line with Deionized Water. 83.5 mg of Ascorbic Acid was quantitatively added to a 50 ml volumetric flask. Using the 2.00 ml volumetric Pipette, pipette 2 ml of the supernatant solution into the 50 ml volumetric flask. Using a graduated cylinder, 20.5 ml of Bipyridyl solution was added to the flask and the solution was filled to the calibration line with the 4.7 pH buffer. The pH of the solution was checked to ensure the pH was near a pH of 5. This second solution was then transferred into a clean and dry plastic storage container. This was then labelled as the unknown solution for later use. A table was created for both the known and the unknown, each detailing in different columns, the volume of original Bipyridyl solution, how much Deionized Water was added, the final volume of the liquid in the test tubes, and the concentration of the liquid in the test tubes. The buret was rinsed with ~5 ml of the Iron Bipyridyl standard and filled with ~20 ml of the standard solution was added. The actual volumes of the standard added were as follows 1.45 ml, 2.23 ml, 3.06 ml, 4.14 ml, and 4.46 ml, each added into a separately cleaned and labelled test tube. The buret was then rinsed with Deionized water and filled to ~25 ml with Deionized Water. The amounts of water added into the test tubes were 6.00 ml, 5.55 ml, 4.45 ml, 3.71 ml, and 3.04 ml. The test tubes were then covered with Parafilm and set aside. A second set of 5 13 x 100 ml test tubes was labelled to indicate that it was used to contain the unknown Iron Bipyridyl solution. The same procedure used to dilute the known solution was applied to dilute the unknown Iron Solution. Respectively, the amounts of Iron solution are: 1.51 ml, 2.29 ml, 3.52 ml, 3.73 ml, and 4.56 ml. The amounts of Deionized water added to the test tubes are as follows: 5.95 ml, 5.40 ml, 4.50 ml, 3.78 ml, and 2.97 ml. A cuvette was obtained, cleaned, and then filled with Deionized Water. The Spectrophotometer was set to 520 nm and the cuvette was inserted, and calibrated this way. The Absorbance and the Percent transmittances were then measured, by emptying the cuvette, rinsing the cuvette in a small amount of the test tube solution, and then adding enough solution after disposing of the rinse liquid to the minimum calibration line. Each sample was done this way and the sample were done in increasing concentration. The actual values and transmittances can be seen later in the report. When all of the Absorbance values were measured for all ten samples, all chemicals and solutions were disposed of in a designated waste container.

**Results (65 points total)**

Split the results into three sections for clarity, and address each part separately. Utilize tables and graphs that are clearly labeled to display the important data and trends in your work.

4A Data and observations (5 points), < 1 page (<500 words)

* Mass of starting material ((NH4)2Fe(SO4)2 • 6H2O) = 2.3990 g
* Color of starting material = a faint pale green, the starting solution with just Sulfuric Acid and Deionized Water was the same color.
* Color changes along the way
  + Yellow precipitate formed when Oxalic Acid was first added to the original solution
  + Prior to decanting, the solution was a clear to faint yellow
  + After washing, the addition of Potassium Oxalate turned the solution into a dirty yellow color
  + The Addition of Hydrogen Peroxide turned the solution into a deep brown color
  + The second addition of Oxalic Acid turned the solution a brilliant green.
  + Small crystals formed as soon as the ethanol solution was added
  + Crystals dissolved upon boiling.
  + After being allowed to settle for 45 hours, the solution formed coarse crystals of about 1 mm in diameter and 2-4 mm long. The crystals were a bright green.
  + The supernatant of the crystals was clear and almost colorless
* Mass, shape, and color of final product - (% Yield is not data, it is a calculation and comes later)
  + The final Mass of the crystals were 2.9009 g
  + They were a bright green, like apple flavored candy. The crystals formed in thin rods of about 2-4 mm in length and about 1 mm in diameter. The crystals

4B Data and observations (5 points), < 1 page (<500 words)

* Quick and dirty titration (not typically used in calculating the average)
  + 0.0968 grams of crystal added
  + 23.36 ml of 0.01000 M KMnO4 solution added to flask
  + The sample turned pink, but did not hold the color, the brown overpowered the pink after less than 30 seconds
  + The sample turned a sludge brown instead of a light pink.
* Three KMnO4 titrations
  + 1
    - 0.0852 g product
    - 18.44 ml of 0.01000 M KMnO4 solution added to flask
  + 2
    - 0.0822 g product
    - 18.92 ml of 0.01000 M KMnO4 solution added to flask
  + 3
    - 0.0898 g product
    - 20.77 ml of 0.01000 M KMnO4 solution added to flask
  + 4 (time and resources permitted a fourth titration to be done, as instructed by the TA for students that had samples with brown ending color instead of pink)
    - 0.0851 g product
    - 20.25 ml of 0.01000 M KMnO4 solution added to flask
* Color changes during the titration
  + Solution started at a light yellow, and turned temporarily brown upon addition of Permanganate solution, but turned pink and clear at the end of the titration. The color changes were the same in all four of the titrations. With the exception of the first “quick and dirty trial”, which was redone, and then the final titration was used in calculating the average.
  + All of the solutions turned brown or some shade of brown after some time.

4C Data and observations (5 points), < 1 page (<500 words)

* Mass of (NH4)2Fe(SO4)2 • 6H2O – 0.1269 g
* Color of Fe(bpy)3 – a bright cherry red in the 50 ml volumetric flask.
* Dilutions of known: 1.45 ml, 2.23 ml, 3.06 ml, 4.14 ml, and 4.46 ml of Known solution with 6.00 ml, 5.55 ml, 4.45 ml, 3.71 ml, and 3.04 ml of Deionized Water added to each test tube respectively
* Wavelength used – 520 nm
* %T/Absorbance data for known

|  |  |  |
| --- | --- | --- |
| Sample | %T | Absorbance |
| 1 | 53.1 | 0.273 |
| 2 | 37.5 | 0.426 |
| 3 | 23.2 | 0.635 |
| 4 | 16.8 | 0.775 |
| 5 | 13.4 | 0.872 |

* Mass of unknown green product – 0.1132 g
* Dilutions: 1.51 ml, 2.29 ml, 3.52 ml, 3.73 ml, and 4.56 ml amounts of Iron solution added with 5.95 ml, 5.40 ml, 4.50 ml, 3.78 ml, and 2.97 ml of water in each test tube respectively
* %T/Absorbance data for unknown green product

|  |  |  |
| --- | --- | --- |
| Sample | %T | Absorbance |
| 1 | 62.4 | 0.206 |
| 2 | 53.4 | 0.257 |
| 3 | 39.4 | 0.405 |
| 4 | 33.8 | 0.471 |
| 5 | 28.4 | 0.546 |

Summary of Data. Include this table in your paper. Fill in the gray areas. (5 points), < 1 page

|  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **4A** | **Starting mass (NH4)2Fe(SO4)2** | | 2.3990 | g |  | **4C** | **Starting mass (NH4)2Fe(SO4)2** | | 0.1269 | g |
|  |  |  |  |  |  |  |  | | | |
|  | **Mass green xtals** | | 2.9009 | g |  |  | **%T known** | **Abs** | Vol. |  |
|  |  |  |  |  |  |  | 53.1 | 0.273 | 1.47 | mL |
| **4B** | **Concentration KMnO4** | | 0.01000 | M |  |  | 37.5 | 0.426 | 2.23 | mL |
|  |  |  |  |  |  |  | 23.2 | 0.635 | 3.06 | mL |
|  | **Trial #1: Mass green xtals** | | 0.0852 | g |  |  | 16.8 | 0.775 | 4.14 | mL |
|  | **Volume KMO4** |  | 18.44 | mL |  |  | 13.4 | 0.872 | 4.46 | mL |
|  |  | | | |  |  |  | | | |
|  | **Trial #2: Mass green xtals** | | 0.0822 | g |  |  | **Mass green xtals** | | 0.1132 | g |
|  | **Volume KMO4** |  | 18.92 | mL |  |  |  |  |  |  |
|  |  | | | |  |  | **% T xtals** | **Abs** | Vol. |  |
|  | **Trial #3: Mass green xtals** | | 0.0898 | g |  |  | 62.4 | 0.206 | 1.51 | mL |
|  | **Volume KMO4** |  | 20.77 | mL |  |  | 53.4 | 0.257 | 2.29 | mL |
|  |  |  |  |  |  |  | 39.4 | 0.405 | 3.52 | mL |
|  |  |  |  |  |  |  | 33.8 | 0.471 | 3.73 | mL |
|  |  |  |  |  |  |  | 28.4 | 0.546 | 4.36 | mL |

Calculations (45 points), < 4 pages (<2000 words)

* Limiting reagent in the synthesis

Iron(II) Ammonium Sulfate is the limiting reagent in the synthesis step because it is the only reagent that is carefully measured and added into the solution used to create crystals. Oxalate cannot be the limiting reagent because there are several points where oxalate ion is added into the solution and all of those points in the experimental ask the student to add the Oxalate to excess, meaning that it cannot be the limiting reagent. The other possible limiting reagent is Potassium, but 7.0 ml of 1.7 M K2C2O4 and the calculation of: x 0.007 L = 0.0238 mol K+.

Clearly, with an empirical formula that calls for three times the number of Potassium Ions as Iron Ions, a simple calculation of 0.0238/0.00611 = 3.89 show s that there are nearly as many as four times as many potassium ions in the solution as Iron ions, meaning that being the proportionally lowest, Iron ion is the limiting reagent in the synthesis step.

* Write the BALANCED redox equation

Redox titration: 2MnO4- + 5 H2C2O4 + 6H+ → 2Mn2+ 10CO­­2 + 8H2O

* Example calculation for moles and % mass of oxalate ligand

In later calculations, the average percent mass of 50.4% of oxalate will be used

* Example calculation of the known Fe(bpy)3 dilutions

* M­1V1=M2V2
* M2 **=** 1.2944 x 10-4 M Fe = Control solution
* M­3 = 2.46 x 10-5 M Fe Known Dilution Sample 1

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Dilution Number  (known) | Volume of Fe2+  Solution (ml) | Volume of DI H2O (ml) | Total  Volume  (ml) | Fe(II) concentration  (mol/L) | %T | Absorbance |
| 1 | 1.47 | 6.00 | 7.47 | 2.46x10-5 | 53.1 | 0.273 |
| 2 | 2.23 | 5.35 | 7.58 | 3.81x10-5 | 37.5 | 0.426 |
| 3 | 3.06 | 4.45 | 7.51 | 5.27x10-5 | 23.2 | 0.635 |
| 4 | 4.14 | 3.71 | 7.85 | 6.83x10-5 | 16.8 | 0.775 |
| 5 | 4.46 | 3.04 | 7.50 | 7.70x10-5 | 13.4 | 0.872 |

* Calibration curve of the known Fe(bpy)3
* Example calculation of the unknown Fe(bpy)3 dilutions

Y = Eb, A = EbC, A = YC 0.546 = 11439C, C = 0.546/11439 = 4.77 x 10-5

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Dilution Number  (unknown) | Volume of Fe2+  Solution (ml) | Volume of DI H2O (ml) | Total  Volume  (ml) | Fe(II) concentration  (mol/L) | %T | Absorbance |
| 1 | 1.51 | 5.95 | 7.46 | 1.80x10-5 | 62.4 | 0.206 |
| 2 | 2.29 | 5.40 | 7.69 | 2.25x10-5 | 53.4 | 0.457 |
| 3 | 3.32 | 4.50 | 7.82 | 3.54x10-5 | 39.4 | 0.405 |
| 4 | 3.73 | 3.78 | 7.51 | 4.12x10- | 33.8 | 0.471 |
| 5 | 4.36 | 2.97 | 7.33 | 4.77x10-5 | 28.4 | 0.546 |

* Example calculation for moles and % mass of Fe
* M2 = 8.02 x 10-5 M 🡪 This is one possible concentration, the average concentration of 8.1 x 10-5 M Fe is used for the following calculations
* M3 = 0.002025 M Fe
* 0.002025 M Fe x 0.100 L Fe = 2.03 x10-4  mol Fe2+
* 2.03 x10-4 mol Fe2+ x = 0.011 g Fe
* Example calculations for Fe:Oxalate ratio
* 10.02% Fe = 10.02 g Fe; 50.4% C2O42- = 50.4 g C2O42-
* ;
* ; 🡪3
* Fe:Oxalate = 1:3
* Example calculations for determination of number of K atoms and K % mass

Fe(C2O4)3 charge = -3; K+ charge = +1; K+ = 3;

0.549 mol K+ x = 21.46% of mass of 100 g sample

* Example calculations for determination of number of H2O molecules and % mass
* 100 g – 21.46 g K+ – 10.02 g Fe – 50.4 g C2O42- = 18.12 g H2O;
* Example calculations for synthesis yield
* 2Fe(NH4)2(SO4)⋅6H2O + 3H2C2O4 + 3K2C2O4 → 2K3[Fe(C2O4)3]⋅3H2O + 4NH4+ + 4SO42- + 6H+ + 6 H2O

= 2.687 g

Yield

Note that the over 100% yield is deceptive, not all of the crystal was necessarily dried to the point where they stopped being over hydrated. The hydrate value of 5 was calculated after the crystals had several days to dry with the foil in the drawer, the experimental value of 2.9009 g of crystals was recorded only 30 minutes after the filtration was complete, meaning that many of the crystals were still “wet” and the trapped water molecules had not had a chance to escape into the atmosphere yet.

**Discussion (40 points)** < 4 pages (<2000 words)

The compound that was synthesized was Potassium Iron(III) Oxalate crystal. This product has the empirical of K3[Fe(C2O4)3] · 5H2O. There is a high confidence that the values for Potassium, Iron, and Oxalate are accurate, based on the calculation of the water hydration state of the crystal and how salts precipitate, the number of water molecules locked within a crystal is dependent not only on the size of the ligands and the complex, but also may vary based on the quality of crystallization. A different crystallization speed may have different numbers of water molecules in the complex. The number of water molecules that are locked in also will change if the sample was baked dry before measurement and analysis.

Of the three titrations, none of them produced any errors, although it is notable that the completed titrations turned a shade of brown after an extended period of time. The titration that gave trouble was the original “quick and dirty” titration that turned brown quickly at the end of the titration instead of holding a light pink color. That titration was redone and the new value was used in later calculations because it was a valid value that actually helped to increase the accuracy of the sample, assuming that the calculated value of K3[Fe(C2O4)3] · 1H2O from post laboratory question 2 is correct. The addition of the new oxalate average brought the average mass percentage from 49% up to 50%, which brought down the number of calculated Oxalate ions down to 3.1, which obviously means that there are 3 Oxalate ions in the empirical formula.

There are many possible sources of error that lead to errors in the calculation. For experiment 4A, there is not much area of error because the crystals were synthesized and the crystals were of the proper physical properties, indicating that there is little error in the synthesis of the complex. The primary source of error would be the procedure where the supernatant was separated from the precipitate. While the supernatant was being decanted, small amounts of the yellow precipitate was lost in the waste container with the initial decanting and every subsequent washing. This is evidenced by the fact that there is not a 100% percent yield in crystals. One other possible source of error is that the weighed initial Iron(II) Ammonium Sulfate crystal weight was not accurate according to the scale and the precision of the scale affected the measured amount of crystal, seeing as the sample added was very small in comparison to the total capacity of the scale, the precision could become a problem, however minor. The last source of error in the final portion of adding the initial crystals, there is a small amount of the crystals that are left on the weighing boat after the thorough cleaning and quantitative transfer.

Experiment 4B involved more areas that could make the error greater. In the beginning, the initial addition of the product into the Erlenmeyer flasks, the weighing boats may have not been properly tared due to time constraints. There may be error originating from not all of the crystals entering the flask with quantitative analysis. The natural uncertainty associated with using a digital device may also affect the uncertainty of the number of crystals that are added to the flasks. Later, during the actual titration, due to the height of the buret, the volume may not have been read accurately and the volume added could have been different from what actually was added. A real possibility in all titrations is that there is overshooting. Overshooting would overestimate the number of oxalate ions that are present in the solution. This was a problem, as evidenced by the fact that the final calculated average composition had 3.1 Oxalate Ions for every Iron(III) ion.

In Experiment 4C, there are many more sources of error than in the other two parts of the experiment. Firstly, the issue with the weighing boats and taring due to time issues. The weighing boats may have had leftover salts on them after thorough quantitative transfer into the volumetric flasks. One other source of error may have been the fact that the liquids added to the volumetric flasks were not at exactly 20° C, which is what the calibration lines of the volumetric flasks were printed and intended to be used with liquids at 20° C, because of this, there may be a slightly lower concentration of the compounds than actually is calculated. This would also apply to the buret used in this portion and the previous Reduction oxidation reaction of the last portion of the experiment. In the transfers, the same applies to the 2 ml volumetric pipette. Although the pipette is guaranteed to work at 20° C, the nature of the inaccuracy of the bulbs used in this lab mean that not exactly 2.00 ml of the solution from the 100 ml volumetric flask was transferred into the 50 ml volumetric flask, meaning that the concentration value is actually undershot. And that would affect the concentration calculations with the unknown because the slope of the line would be steeper than it should be, meaning that all of the calculations would indicate the actual concentration of the Iron is lower than the calculations would suggest. The glassware issues in this portion of the lab create systematic errors into calculations, because the liquids are constantly being transferred from one container to the next, it is difficult to calculate and quantitatively account for the amount of lost iron ions because in order to account for the heat expansion of glass, the heat expansion constant of each piece of glassware would be needed for calculations of tiny amounts of lost fluid. It should be noted that in a more concentrated solution, this would be a smaller problem because the number of ions in a milliliter would be thousands of times higher. The fact that these pieces of glass ware and this lad deal in millimoles means that every drop would actually account for a significant amount of ions in comparison to the volume of lost liquid. Another source of error is that during transfer of the 2 ml of iron solution into the 50 ml volumetric flask, there were small amounts of Iron solution stuck in the volumetric pipette, even after attempts to remove it. This lost Iron would mean that the concentration of Iron is again, even higher than what it actually should be. Even if all of these errors were to be accounted for, the concentration of the solution would still only be affected by a tiny amount, possibly only the third and smallest significant figure being affected out of three significant figures.

While there is little literature in the way of synthesis of the Iron(III) oxalate complex literature, there are several applications of the complex that seem to be of note. Firstly, because of the fact that the substance and its derivatives are light sensitive, it is used often as a coordination complex for teaching concepts of coordination chemistry in pigments for students. (Fiorito and Polo 1). The light sensitive properties of this substance allows different atomic spectra in the blue range to be reflected. This is the reason that this compound can be applied in cyanotype printing, which prints in blue (which is coincidentally one of the colors of the compounds that this complex decays into), or more commonly, Prussian blue. The usage of this is similar to silver nitrate, which was used in legacy style photography to stain high quality paper with photographs.

In terms of issues with performing the work, the main problem that was a barrier to accuracy was time constraint. There was no way to dry the crystals, so weighing the crystals right after the filtration lead to overshooting, in the case of this particular experiment, the lack of drying was so extreme that it led to having a yield percentage of over 100%. It is recommended for later repeats of this lab to set aside time to dry the crucibles and the sample to remove as much water as possible from the crystals as possible. Next, for the portion in experiment 4C that involves the centrifuge, the centrifuge time should be longer to create a more stable pellet and waste less time in general, as a person who needed to have a solution re-centrifuged would generally have to wait for others to go once, meaning that the time used waiting is squandered, and having a longer centrifuge time would waste less time in transferring between centrifuges and people, increasing efficiency.

Iron(III) Oxalate is a metal ion that is crystalized with the help of the ligand Oxalate. This sort of “precipitation” was also seen in Experiment 1. This lab employed many lab techniques from previous labs, such as gravimetric analysis for yield percentage, volumetric analysis for determining the number of Oxalate ion in the sample, and spectrophotometry for analysis of the iron content of the crystals.

**References (5 points), no limit on number of pages**

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