

Experiment 4: Synthesis and analysis of iron oxalate

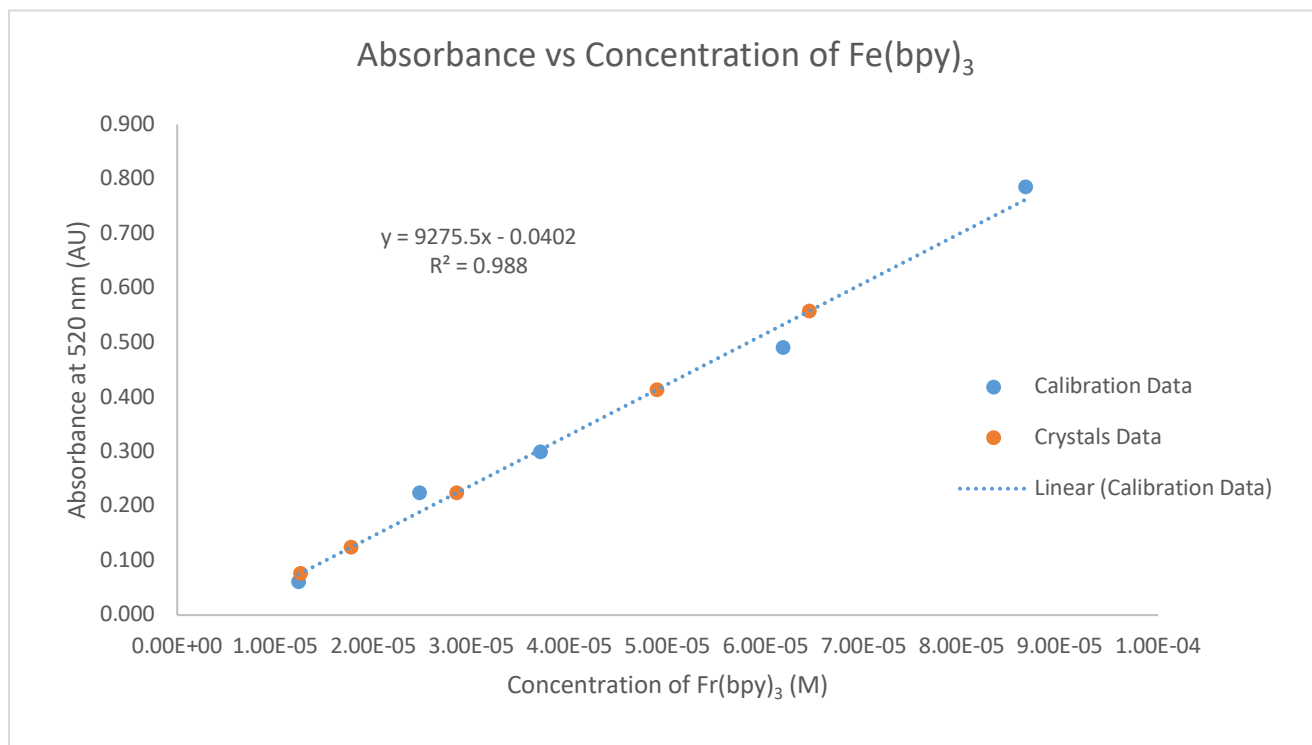
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Data (10 pts):

Part I				Starting (NH ₄) ₂ Fe(SO ₄) ₂ (~1.2 g)		1.3330		g			
Mass green crystals after filtration						1.0776		g			
Parts II-IV						(NH ₄) ₂ Fe(SO ₄) ₂ for UV-Vis (~0.12 g)		0.1211		g	
Standard		%T standard	Abs standard		Vol. aliquot		Vol. final		Standard Fe ²⁺ concentration		
dilution 1		86.8	0.0615		0.75 mL		7.50 mL		1.24E-5		M
dilution 2		59.7	0.224		1.50 mL		7.50 mL		2.47E-5		M
dilution 3		50.1	0.300		2.25 mL		7.50 mL		3.71E-5		M
dilution 4		32.3	0.491		3.75 mL		7.50 mL		6.18E-5		M
dilution 5		16.4	0.785		5.25 mL		7.50 mL		8.65E-5		M
Mass green crystals for UV-Vis (~0.12 g)						0.1211		g			
Crystals		% T crystals	Abs crystals		Vol. aliquot		Vol. final				
dilution 1		83.8	0.0768		0.75 mL		7.50 mL				
dilution 2		75.1	0.124		1.50 mL		7.50 mL				
dilution 3		59.7	0.224		2.25 mL		7.50 mL				
dilution 4		38.6	0.413		3.75 mL		7.50 mL				
dilution 5		27.7	0.558		5.25 mL		7.50 mL				

Data (16 pts):

Linear fit gives (also input equation into Canvas):

(report at least 5 sig figs for m and b)

$$y = mx + b = 9275.5x - 0.040164$$

Resulting iron concentrations:

Xtals	Xtals Fe^{2+} concentration	
dilution 1	1.26E-5	M
dilution 2	1.77E-5	M
dilution 3	2.85E-5	M
dilution 4	4.89E-5	M
dilution 5	6.44E-5	M

Results:

- (A) Mass % of Fe (two decimal places): $11.52\% \pm 1.73\%$
- (B) Mass % of oxalate (C_2O_4) is provided: **53.75%**
- (C) The compound you synthesized has the following empirical formula: $K_w[Fe_x(C_2O_4)_y] \cdot zH_2O$.
Based on the mass %s of Fe and oxalate from parts (A) and (B) above, report the number of moles of Fe (x) and C_2O_4 (y) if you assume a mass of 100 g of the compound $K_w[Fe_x(C_2O_4)_y] \cdot zH_2O$.
Report the values with two decimal places:
In 100 g, the number of moles of Fe (a) and C_2O_4 (b): $a = 0.21$ $b = 0.61$
- (D) Report the empirical formula with the smallest integer values for $K_w[Fe_x(C_2O_4)_y] \cdot zH_2O$ by stating the integer molar equivalents for K (w), Fe (x), C_2O_4 (y), and H_2O (z) in the spaces below.
There should be at least one value of 1 in the empirical formula.
- $w = 3$ $x = 1$ $y = 3$ $z = 3$
- (E) Molar mass based on your empirical formula from part (D): 491.24 g/mol (two decimal places)
- (F) Yield of synthesis based on empirical formula from part (D): 64.53% (two decimal places)

Assumptions (15 pts):

1. One assumption that was made in this experiment was that the recrystallization boiling process was carried out without EtOH evaporation. In the process of recrystallizing the crystals, the solution was boiled, allowing the impure crystals to disassociate back into solution. However, during this process, some of the EtOH evaporated. The EtOH was added to the solution to help crystals form, so removing some by evaporation could've resulted in less crystals being formed. This would ultimately lead to an artificially reduced yield but would not affect the %mass or empirical formula because those calculations were done to scale with the amount of crystal collected.
2. Another assumption that was made in this experiment was assuming that there was no localized heating while adding H_2O_2 . If there was localized heating above $50^\circ C$ in any part of the solution, the hydrogen peroxide would be destroyed, leaving the solution with less hydrogen peroxide, leading to incomplete oxidation of Fe^{2+} to Fe^{3+} if not enough peroxide was present to fully react with the Fe^{2+} . This would then lead to the formation of the $Fe^{2+}(OX)$ precipitate. This impure precipitate has a higher % mass Fe than the Fe^{3+} solution. This is problematic because it would lead to the calculated mass % Fe of the Fe^{3+} precipitate to be higher if the assumption was not valid.
3. Another assumption made in this experiment was assuming that all the free Fe^{3+} supernatant had been collected after centrifuging down the $CaOx$ precipitate. Although the precipitate was washed a total of three times, there still could have been residual supernatant that was left behind, meaning not all the of the Fe^{3+} in the collected crystals would be used. This would lead to a reduced % mass Fe because there would be less Fe in the diluted solutions resulting in a higher absorbance value, causing the calculated % mass Fe to be smaller.

Experimental (21 pts).

To begin the formation of the Fe(II)-oxalate compound, DI water was brought to a gentle boil in a 250 mL beaker using a hot plate set at 350°C. Five boiling chips were also placed in the water to ensure a calm boil. Once a gentle boil was reached, 1.3330 g of $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ was placed in a clean 50 mL beaker along with 3 mL of DI water. Once the $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ was dissolved, two drops of 6 M sulfuric acid was carefully added to the solution. Then, 6.00 mL of 1M oxalic acid was very carefully added to the solution. A orange-yellow precipitate formed at the bottom of the solution, and pH paper was used and found a pH of 4. More sulfuric acid was added until the pH reached 1 and was a vibrant orange-red. The beaker was then placed in the gentle boiling water bath for about five minutes, allowing the precipitate to fully form. Using a clamp, the beaker was angled at a 45° angle, allowing the precipitate to settle. The supernatant was then decanted completely and placed in a waste beaker. The precipitate was then washed three times with 4 mL of hot DI water (80°C). Each time, the supernatant was decanted and placed in the waste beaker. The Fe(II)-oxalate compound is what remained.

To oxidize the Fe(II)-oxalate solution to Fe(III)-oxalate, 3.00 mL of 1.7 M potassium oxalate was first added to the washed precipitate. A stir bar was placed into the solution and set at 150 rpm. A temperature probe was also added to ensure the solution stayed at about 40°C. To further ensure the solution wouldn't exceed the allowed temperature range, the solution was held 6-10 cm above the hotplate using a clamp. 10.00 mL of 6% H_2O_2 was added to a graduated cylinder and was then slowly added dropwise to the solution very slowly. The hydrogen peroxide was added slowly because the reaction is exothermic, meaning it produces heat. To prevent large scale decomposition of the hydrogen peroxide, the bulk temperature of the solution was kept between 38°C and 44°C. The solution approached a dark brownish color as more hydrogen peroxide was added. Once the full 10.00 mL of hydrogen peroxide was added to the solution, a brown solid remained at the bottom of the beaker. The solution was then heated until it reached a boil and was kept there for about 10 seconds. To dissolve the brown solid, 2.0 mL of 1 M oxalic acid was added. The solution was then stirred for a few minutes until the solution turned a shade of light brown. Another 0.8 mL of oxalic acid was added to the solution and stirred until the solution was a yellowish green. The iron is now in the Fe^{3+} oxidation state.

Once the solution cooled to room temperature, 10.00 mL of 95% EtOH (ethanol) was added. Precautions were taken to ensure the ethanol did not go near the hot plate because it is flammable. A light green precipitate formed on contact with the ethanol. The solution was then added back to the hot plate and brought to a boil for 10 seconds to redissolve the impure crystal precipitate that was formed. The solution was then left to sit until it cooled and then wrapped with parafilm and aluminum foil to block light from hitting the solution. It was stored in a dark environment where the pure crystal precipitate could form. After the crystals had formed a few days later, vacuum filtration was run on the solution to isolate the crystals from the liquid in the solution. During the vacuum filtration, the crystals were washed with 5.00 mL ethanol twice instead of DI water because the crystals are soluble in water. The crucible with the crystals was vacuumed for 10 minutes until dry and then left to sit for another 30 minutes to dry before being massed.