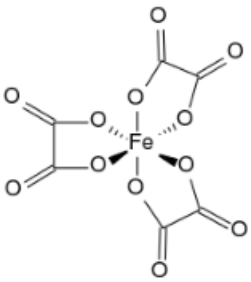
CEAC 103
GENERAL CHEMISTRY

Experiment 4

Preparation and Analysis of Potassium Trisoxalatoferrate(III) Trihydrate, $K_3[Fe(C_2O_4)_3].3H_2O$



Purpose: To prepare the complex trisoxalatoferrate(III), $Fe(C_2O_4)_3^{-3}$ anion and isolate it as its hydrated potassium salt, $K_3[Fe(C_2O_4)_3].3H_2O$. Also, to study the photochemical reduction of the sample.

APPARATUS AND CHEMICALS:

$K_2C_2O_4.H_2O$ (Potassium oxalate monohydrate)	filter paper
$FeCl_3.6H_2O$ (Iron (III) chloride hexahydrate)	distilled water
$K_3Fe(CN)_6$ solution (Potassium hexacyanoferrate(III))	funnel
H_2SO_4 solution (Sulfuric acid)	100-mL beaker
test tubes	

THEORY:

Potassium trisoxalatoferrate(III) trihydrate, $K_3[Fe(C_2O_4)_3].H_2O$ is a green crystalline salt, soluble in hot water but rather insoluble when cold. It can be prepared by the reaction of $K_2C_2O_4.H_2O$ with $FeCl_3.6H_2O$.

$$3K_2C_2O_4.H_2O(aq) + FeCl_3.6H_2O(aq) \rightarrow K_3Fe(C_2O_4)_3].3H_2O(aq) + 3KCl(aq)$$

The complex anion is photo-sensitive. This means that upon exposure to light of an appropriate wavelength (<450 nm in this case) the $Fe(C_2O_4)_3^{-3}$ undergoes an intramolecular redox reaction in which the Fe(III) anion is reduced to Fe(II) while one of the oxalate groups is oxidized to CO_2.

$$[Fe(C_2O_4)_3]^{3-} \rightarrow Fe^{2+} + 5/2\ C_2O_4^{2-} + CO_2(g)$$

As mentioned above, light causes an internal electron-transfer reaction to occur in $[Fe(C_2O_4)_2]^{3-}$ ion, producing CO_2 and Fe^{2+} ions. The Fe^{2+} that is produced can readily be detected by adding a solution of potassium ferricyanide $K_3Fe(CN)_6$. A deep blue colored ferroferri cyanide complex is formed.

$$Fe^{2+} + Fe(CN)_6^{3-} \rightarrow Fe[Fe(CN)_6]^{-}$$

ferroferricyanide deep blue.

PROCEDURE:

A. Preparation of $K_3[Fe(C_2O_4)_3].3H_2O$

1. Weigh approximately 9.0 g of hydrated potassium oxalate, $K_2C_2O_4.H_2O$ into a 250 mL beaker.
2. Add 30 mL of distilled water and heat to dissolve (do not boil).
3. In a second small beaker dissolve 4.4 g of $FeCl_3.6H_2O$ in a minimum amount of cold water (10-15 mL). Add the $FeCl_3.6H_2O$ solution to the warm oxalate solution and stir with a glass rod. Allow the product to crystallize (away from strong sunlight) by cooling the solution in an ice-water mixture.
4. Collect the crystalline product by filtration. The product is $K_3[Fe(C_2O_4)_3].3H_2O$.

B. Blueprinting

1. Wet a piece of filter paper with $[Fe(C_2O_4)_2]^{3-}$ solution.
2. Leave it to dry. (Meanwhile you can follow part C)
3. Place small opaque objects (coins, keys, etc.) on the paper.
4. Irradiate for few minutes using a light source (If not available you may use bright sunlight)
5. Dip the paper into potassium ferricyanide solution (CAUTION potassium ferricyanide is poisonous. Avoid contact with your skin. If it happens immediately wash your skin with plenty of water.)
6. Remove the developed blueprint and dip in a beaker of distilled water to wash off excess ferricyanide solution. Explain your observations.

C.Photochemical Reaction of $[Fe(C_2O_4)2]^{3-}$

1. Dissolve 0.7 g of your complex in 100 mL of distilled water in a flask. Add 3 mL of 2 M H_2SO_4 and swirl the mixture. To each three labeled test tubes add 10 mL of this solution.
2. Keep one tube away from the light source as the control and irradiate the remaining two tubes with the light source for 1 and 5 minutes respectively.
3. To all three tubes add 1 mL of 0. 1 M potassium ferricyanide solution $K_3Fe(CN)_6$.
4. Record and explain your observations.

DATA SHEET

Preparation and Analysis of Potassium Trisoxalatoferrate(III) Trihydrate, $K_3[Fe(C_2O_4)_3].3H_2O$

Student's Name : Date:

Laboratory Section/Group No :

Assistant's Name and Signature :

A .Blueprinting

Observations:

Explain:

B.Photochemical Reaction of $[Fe(C_2O_4)_2]^{-3}$

Observations:

1st sample:

2nd sample:

3rd sample: