Experiment 6: Preparation and Analysis of Potassium Trisoxalatoferrate(III) Trihydrate, K₃[Fe(C₂O₄)₃].3H₂O

PURPOSE:
To prepare the complex trisoxalatoferrate(III), Fe(C₂O₄)₃⁻³ anion and isolate it as its hydrated potassium salt, K₃[Fe(C₂O₄)₃].3H₂O. Also, to study the photochemical reduction of the sample.

APPARATUS AND CHEMICALS:
K₂C₂O₄.H₂O
FeCl₃.6H₂O
K₃Fe(CN)₆ solution
H₂SO₄ solution
distilled water
funnel
filter paper
100-mL beaker
test tubes
opaque objects

THEORY:
Potassium trisoxalatoferrate(III) trihydrate, K₃[Fe(C₂O₄)₃].H₂O is a green crystalline salt, soluble in hot water but rather insoluble when cold. It can be prepared by the reaction of K₂C₂O₄.H₂O with FeCl₃.6H₂O.

\[
3K₂C₂O₄.H₂O(aq) + FeCl₃.6H₂O(aq) \rightarrow K₃Fe(C₂O₄)₃].3H₂O(aq) + 3KCl(aq)
\]

The complex anion is photo-sensitive. This means that upon exposure to light of an appropriate wavelength (<450 nm in this case) the Fe(C₂O₄)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 CO₂.

\[
[Fe(C₂O₄)₃]\text{⁻³} \rightarrow Fe^{2+} + 5/2 C₂O₄^{2⁻} + CO₂(g)
\]

As mentioned above, light causes an internal electron-transfer reaction to occur in [Fe(C₂O₄)₂]³⁻ ion, producing CO₂ and Fe²⁺ ions. The Fe²⁺ that is produced can readily be detected by adding a solution of potassium ferricyanide K₃Fe(CN)₆. A deep blue colored ferroferri cyanide complex is formed.

\[
Fe^{2⁺} + Fe(CN)₆^{3⁻} \rightarrow Fe[Fe(CN)₆]^\text{⁻²}
\]

ferroferricyanide deep blue.
PROCEDURE:

A. Preparation of \( \text{K}_3[\text{Fe(C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O} \)

1. Weigh approximately 9.0 g of hydrated potassium oxalate, \( \text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O} \) 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 \( \text{FeCl}_3 \cdot 6\text{H}_2\text{O} \) in a minimum amount of cold water (10-15 mL). Add the \( \text{FeCl}_3 \cdot 6\text{H}_2\text{O} \) 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 \( \text{K}_3[\text{Fe(C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O} \).

B. Blueprinting

1. Wet a piece of filter paper with \( \text{[Fe(C}_2\text{O}_4)_2]^3- \) 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 \( \text{[Fe(C}_2\text{O}_4)_2]^3+ \)

1. Dissolve 0.7 g of your complex in 100 mL of distilled water in a flask. Add 3 mL of 2 M \( \text{H}_2\text{SO}_4 \) 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 \( \text{K}_3\text{Fe(CN)}_6 \).
4. Record and explain your observations.
DATA SHEET

Preparation and Analysis of Potassium Trisoxalatoferrate(III) Trihydrate, K$_3$[Fe(C$_2$O$_4$)$_3$].3H$_2$O

Student's Name : Date:
Laboratory Section/Group No :
Assistant's Name and Signature :

B. Blueprinting

*Observations:*

*Explain:*

C. Photochemical Reaction of [Fe(C$_2$O$_4$)$_2$]$^{3-}$

*Observations:*

1st sample:

2nd sample:

3rd sample:

*Explain*