EXPERIMENT

Aim

To prepare a pure sample of the complex potassium ferric oxalate $K_3[Fe(C_2O_4)_3].3H_2O$.

Theory

The complex potassium trioxalatoferrate (III) can be prepared by dissolving freshly prepared ferric hydroxide in a solution of potassium oxalate and oxalic acid.

$$FeCl_3 + 3KOH \longrightarrow Fe(OH)_3 + 3KCl$$

$$2Fe(OH)_3 + 3\begin{matrix}COOH\\|\\COOH\end{matrix} + .2H_2O \longrightarrow Fe_2(C_2O_4)_3 + 12H_2O$$

$$Fe_2(C_2O_4)_3 + 3\begin{matrix}COOK\cdot H_2O\\|\\COOK\end{matrix} \longrightarrow 2K_3[Fe(C_2O_4)_3]\cdot 3H_2O$$

Other names – Potassium trioxalatoferrate (III), Potassium ferrioxalate, Potassium iron (3+) oxalate, Potassium tris(oxalate) ferrate (III).

Material Required

Three beakers (250 mL), China dish, funnel, funnel-stand, glass-rod, wash bottle, tripod stand and wire gauze. Ferric chloride, oxalic acid hydrated, potassium oxalate and potassium hydroxide.

Procedure

1. Dissolve 3.5 g of anhydrous ferric chloride and 50 mL of distilled water in a 250 mL beaker.
2. In another beaker dissolve 4 g of potassium hydroxide in 50 mL of water.
3. Add KOH solution to $FeCl_3$ solution in small portions with constant stirring. Filter the precipitates of ferric hydroxide so formed through a Buchner funnel. Wash the ppt. with distilled water.
4. In another beaker (250 mL) take 4 g of hydrated oxalic acid and 5.5 g of hydrated potassium oxalate. Add about 100 mL of water and stir thoroughly to get a clear solution.
5. Add the freshly prepared $Fe(OH)_3$ ppt. in small amounts to the above solution with constant stirring. The ppt. gets dissolved. If ppt. does not dissolve, then warm it and leave the contents for some time.
6. Filter and transfer the filtrate to China dish and heat on a sand bath or wire-gauze to obtain crystallization point.
7. Now place the China dish on a beaker full of cold water and keep it aside for crystallization. China dish should be covered with a black paper as the complex is sensitive to light.
8. Decant off the mother liquor, wash the crystals with a small amount of ethyl alcohol. and dry them between the folds of filter paper.
9. Find out the weight of the crystals.

Observations

Weight of the crystals obtained = ... g

Color of the crystals is .....

Result

The yield of Potassium ferric oxalate is _____ gm.

Precautions

1. Do not concentrate on the solution too much.
2. Let the concentrated solution cool slowly and undisturbed to get large crystals.

VIVA VOCE

Q 1. Why is potassium ferric oxalate considering a complex compound?

Ans. It is a complex compound because it contains a central metal ion (Fe^{3+}) coordinated with oxalate ions ($C_2O_4^{2-}$) and water molecules.

Q 2. What role does oxalic acid play in the preparation process?

Ans. Oxalic acid acts as a ligand, coordinating with the ferric ions to form the complex.

Q 3. How is the purity of potassium ferric oxalate ensured during the preparation?

Ans. Purity is ensured by using pure reagents, avoiding contamination, and employing proper laboratory techniques. Recrystallization may also be used.

Q 4. Discuss the significance of the three water molecules in the formula $K_3[Fe(C_2O_4)_3]\cdot 3H_2O$.

Ans. The water molecules are part of the coordination sphere, and their presence helps stabilize the complex structure.

Q 5. How can you confirm the presence of ferric ions in the prepared complex?

Ans. Ferric ions can be confirmed using chemical tests like the formation of a reddish-brown precipitate with thiocyanate ions.

Q 6. Explain the color change observed during the reaction for potassium ferric oxalate preparation.

Ans. The color change is due to the formation of the ferric oxalate complex, which often has a distinct color.

Q 7. Discuss the stoichiometry of the reaction involved in the preparation.

Ans. Provide a balanced chemical equation for the reaction, highlighting the molar ratios of reactants and products.

Q 8. Discuss the applications or uses of potassium ferric oxalate.

Ans. Potassium ferric oxalate finds applications in photography and actinometry for determining light intensity.