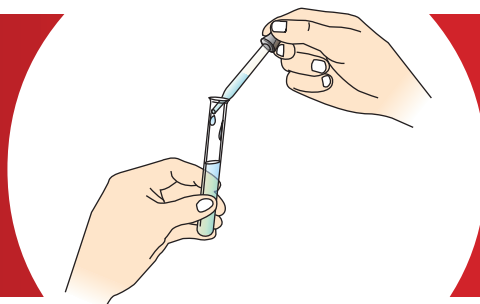


UNIT-9

PREPARATION OF INORGANIC COMPOUNDS



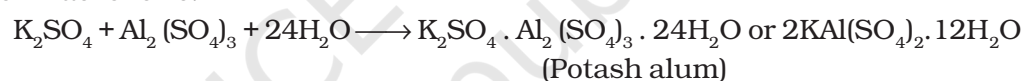
EXPERIMENT 9.1

Aim

To prepare double salts: ferrous ammonium sulphate (Mohr's salt) and potash alum.

Theory

When a mixture containing equimolar proportions of potassium sulphate and aluminium sulphate or ferrous sulphate and ammonium sulphate is crystallised from its solution, a double salt is formed. The formation of double salt may be shown as follows:



Ferrous ammonium sulphate (Mohr's salt)

Fe^{2+} and Al^{3+} ions undergo hydrolysis, therefore, while preparing aqueous solutions of ferrous sulphate and aluminium sulphate in water, 2-3 mL dilute sulphuric acid is added to prevent the hydrolysis of these salts.

Material Required



- Beaker (50 mL) : One
- Conical flask (50 mL) : One
- Trough : One
- Glass rod : One
- Tripod stand : One
- Funnel : One
- Wire gauze : One



- Potassium sulphate : As per need
- Aluminium sulphate : As per need
- Ferrous sulphate : As per need
- Ammonium sulphate : As per need
- Dilute sulphuric acid : As per need
- Ethanol : As per need

Procedure

(a) Preparation of Double Salt: Potassium Aluminium Sulphate (Potash Alum)

- (i) Take 10 mL of distilled water in a 50 mL beaker and heat it to about 40°C. Dissolve 6.6 g of aluminium sulphate in it and add about 0.4 mL of dilute sulphuric acid.

- (ii) Weigh 2.4 g of powdered potassium sulphate and transfer it to the above solution.
- (iii) Heat the solution with constant stirring till potassium sulphate dissolves completely.
- (iv) Allow the solution to cool to room temperature slowly.
- (v) On cooling, white crystals of potash alum separate out.
- (vi) Decant the mother liquor and wash the crystals by gently shaking with 1:1 cold water and alcohol mixture.
- (vii) Filter the crystals, dry these between the folds of a filter paper and note the yield.

Ethanol



Sulphuric acid



(b) Preparation of Double Salt: Ferrous Ammonium Sulphate

- (i) Dissolve 3.5 g of ferrous sulphate and 1.7 g of ammonium sulphate (weighed separately), in 5 mL of distilled water contained in a 50 mL conical flask by heating. Add about 0.5 mL of dilute sulphuric acid to the flask and concentrate the solution by heating till the crystallization point is reached.
- (ii) Allow the mixture to cool to room temperature slowly.
- (iii) On cooling, light green crystals of ferrous ammonium sulphate separate out.
- (iv) Decant the mother liquor and wash the crystals by shaking with very small amounts of 1:1 cold water and alcohol mixture to remove sticking mother liquor.
- (iv) Separate the crystals by filtration wash with alcohol, dry between the folds of a filter paper and record the yield.

Result

Yield of potash alum/Mohr's salt is _____ g.

Precautions

- (a) Cool the solution slowly to get good crystals. Avoid rapid cooling.
- (b) Do not disturb the solution while cooling.
- (c) Avoid prolonged heating while preparing crystals of ferrous ammonium sulphate, as it may oxidise ferrous ions to ferric ions and change the stoichiometry of the crystals.



Discussion Questions

- (i) Why do we take equimolar quantities of reacting compounds in the preparation of double salts?
- (ii) In the preparation of ferrous ammonium sulphate, can concentrated sulphuric acid be used in place of dilute sulphuric acid? Explain.
- (iii) What is the difference between iron compounds; $K_4[Fe(CN)_6]$ and $FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$?

- (iv) What is the action of heat on potash alum?
- (v) Why does an aqueous solution of potash alum turn blue litmus red?
- (vi) What are the isomorphous substances?
- (vii) Give the name of some of the alums where cations are other than Al^{3+} .
- (viii) What is the difference between a complex compound and a double salt?

EXPERIMENT 9.2

Aim

To prepare potassium trioxalatoferate(III).

Theory

When hydrated ferric chloride is dissolved in aqueous solution of oxalic acid containing potassium hydroxide, green crystals of potassium trioxalatoferate(III) are obtained. The reaction involved in the formation of these green crystals is as follows:



Material Required



- Beaker (50 mL) : One
- Porcelain dish : One
- Water bath : One
- Glass rod : One
- Funnel : One



- Ferric chloride : 2.5 g
- Potassium hydroxide : 3.8 g
- Oxalic acid : 3.0 g
- Ethanol : As per need

Procedure

Potassium hydroxide



Ethanol



Oxalic acid



- (i) Prepare a solution of 3.0 g of oxalic acid in 12.5 mL of hot water contained in a clean 50 mL beaker.
- (ii) To the above solution, add 3.8 g of potassium hydroxide gradually in lots, with stirring so that it dissolves completely.
- (iii) Add 2.5 g of ferric chloride into the above solution with constant stirring till it is completely dissolved.
- (iv) Filter the solution and concentrate the green filtrate by heating in a porcelain dish over a water bath and cool the mixture slowly.
- (v) Filter the crystals so formed, wash with 1:1 mixture of cold water and alcohol and dry them by pressing between the folds of a filter paper.

Result

Yield of potassium trioxalatoferrate (III) is _____ g.

 **Precautions**

- (a) Do not evaporate the entire solvent when the solution is being concentrated.
- (b) Weigh the desired quantities of different substances accurately.
- (c) Maintain the temperature of hot water around 40°C.
- (d) Add potassium hydroxide to oxalic acid solution in small lots.

**Discussion Questions**

- (i) Give IUPAC name of the compound called potassium ferrioxalate.
- (ii) What is the coordination number of iron in potassium trioxalatoferrate(III)?
- (iii) Give two examples of bidentate ligands other than oxalate ion.
- (iv) Why does the compound, potassium trioxalatoferrate(III), not give tests for ferric ions?
- (v) What are chelates ?