S.Y.B.SC. INORGANIC CHEMISTRY

PRACTICAL SEM-II

ONLINE LECTURE NO. 2

INORGANIC PREPARATIONS

# DATE:- 17, MAY 2021

TIME: (12.30 P.M.)

A salt is formed by the neutralization of an acid by a base. There are different types of salts. They are:

1) Simple salt: $KOH + HCl \rightarrow KCl + H2O$

2) Molecular (or) addition compounds:

a) Double salts are molecular compounds which are formed by the evaporation of solution containing two (or) more salts in stoichiometric proportions. Hence the molecular compounds which dissociate in solution into its constituent ions are known as double salt. Double salts retain their properties only in solid state. They are also called as lattice compounds. **Example** Mohr's salt:

$$K_2SO_4 \cdot Al_2(SO_4)_3 \cdot 6H_2O \rightarrow 2K^+ + 2Al^{3+} + 4SO_4^{2-} + 6H_2O^{\circ}$$
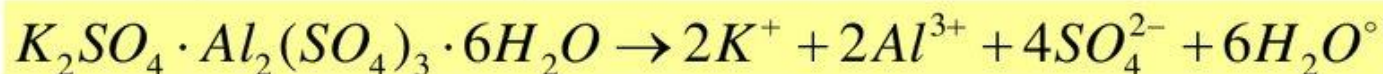
b) Coordination (or complex) compounds is 'a compound formed from a Lewis acid and a Lewis base'. The molecular compounds, do not dissociate into its constituent ions in solution are called coordination compounds.

$$Fe(CN)_2 + 4KCl \rightarrow K_4[Fe(CN)_6] \quad or \quad Fe(CN)_2 \cdot 4KCl$$

$$K_4[Fe(CN)_6] \rightarrow 4K^+ + [Fe(CN)_6]^{4-}$$
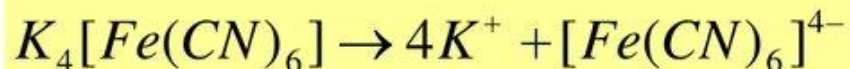
Ferrous cyanide

Ligand: Electron rich species having tendency to donate electrons

N (7) $\rightarrow$ $1S^2$, $2S^2$, $2P_x^1$ $2P_y^1$ $2P_z^1$

H2N-CH2-CH2-NH2
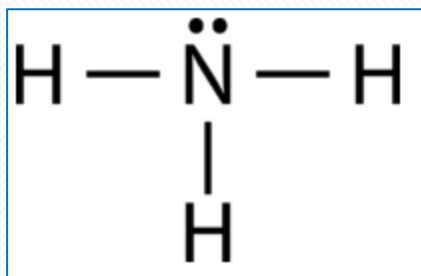
Chelate
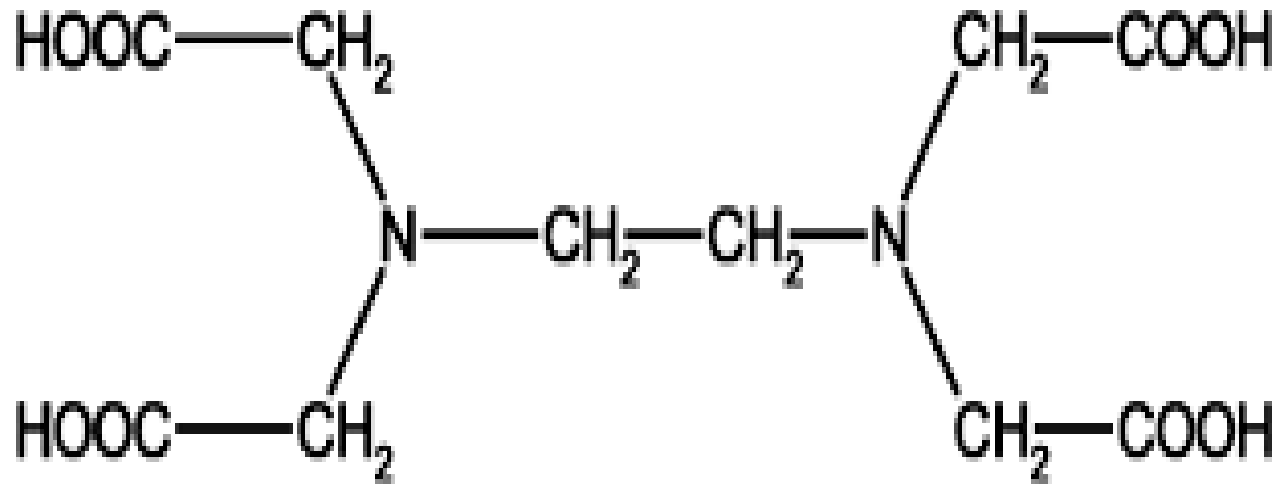
Figure 1. Molecular structure of EDTA

$$H:\overset{\cdot\cdot}{\underset{\cdot\cdot}{N}}: \; + \; \overset{F}{\underset{F}{B}:F} \longrightarrow H:\overset{H}{\underset{\cdot\cdot}{N}}:\to \overset{F}{\underset{F}{B}}:F \longrightarrow H-\overset{H}{\underset{H}{N}} \to \overset{F}{\underset{F}{B}}-F$$
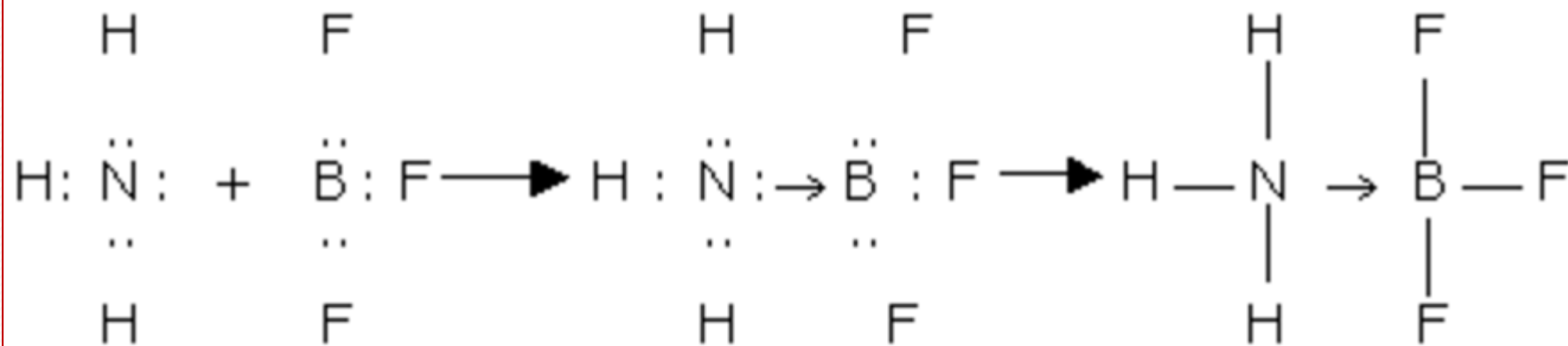
Difference between Double Salt and Complex Compound

Double Salt	Complex Compound
They do not have coordinate bonds.	They have coordinate bonds.
They exist in solid state.	They exist in solid state as well as in aqueous solutions.
They lose their identity in aqueous solution.	They do not lose their identity in aqueous solution.
They contain two salts in equimolar ratio.	They contain ions which may or may not be in equimolar ratio.

DOUBLE SALT
VERSUS
COMPLEX SALT

A double salt is a compound prepared by the combination of two different salt compounds	A complex salt is a compound composed of a central metal atom having coordination bonds with ligands around it
Completely dissociate into its ions in water	Do not completely dissociate into its ions in water
Give simple ions when added to water	Do not give simple ions
Can easily be analyzed by determining the ions present in the aqueous solution	Cannot be easily analyzed by determining the ions in the aqueous solution

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Double Salts	Complex Compound
➤ They usually contain two simple salts in equimolar proportions.	➤ The simple salts from which they are formed may or may not be in equimolar proportions.
➤ They exist only in the solid state. In aqueous solution, they dissociate completely into ions.	➤ They exist in the solid state as well as in aqueous solutions. This is because even in the solution, the complex ion does not dissociate into ions.
➤ They are ionic compounds and do not contain any coordinate bond.	➤ They may or may not be ionic but the complex part is always contains coordinate bonds.
➤ The properties of the double salt are the same as those of its constituent compounds.	➤ The properties of the coordination compound are the different from its constituents.
➤ In the double salts, the metal ions show their normal valency.	➤ In a coordination compound, the metal ions satisfy its 2 types of valencies called primary and secondary valencies.
➤ A double salt loses its identity in the solution.	➤ A coordination compound retains its identity in its solution.

On the basis of nature, addition (or) molecular compoun
are divided into two categories. They are double salts a
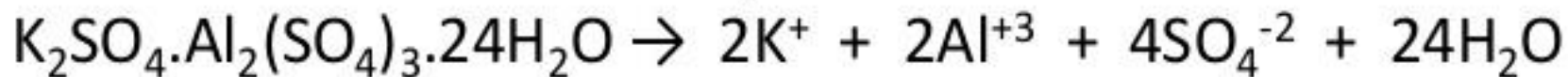coordination (or) complex compounds.

differences between double salt and co-ordination compound.

Double Salt	Co-ordination compound
1. These exist only in solid state and dissociate into constituent species in their solution.	1. They retain their identity in solid as well as in solution state.
2. They lose their identity in dissolved state.	2. They do not lose their identity in dissolved state.
3. Their properties are essentially the same as those of constituent species.	3. Their properties are different from those of their constituents. For example $K_4[Fe(CN)_6]$ does not show the test of Fe^{2+} and CN^- ions.
4. In double salts the metal atom/ion exhibit normal valency.	4. In co-ordination compounds, the number negative ions or molecules surrounding the central metal atom is beyond its normal valency.

Mohr's salt: $FeSO_4.(NH_4)_2SO_4.6H_2O$
double salt.

Double salt and coordination compound

- Ex: An aqueous solution of potash alum will give the tests for K^+, Al^{+3}, and SO_4^{-2}

$$K_2SO_4.Al_2(SO_4)_3.24H_2O \rightarrow 2K^+ + 2Al^{+3} + 4SO_4^{-2} + 24H_2O$$

- On the other hand, coordination compounds are molecular compounds that retain their identity even when dissolved in water.

- Ex: When potassium ferrocyanide is dissolved in water, it does not give the usual tests for Fe^{+2} and CN^{-1}, indicating that, $[Fe(CN)_6]^{-4}$ does not dissociate into Fe^{+2} and CN^{-1}.

$$K_4[Fe(CN)_6] \longrightarrow 4K^+ + [Fe(CN)_6]^{-4}$$

$$[Fe(CN)_6]^{-4} \xrightarrow{\quad X \quad} Fe^{+2} + 6CN^-$$
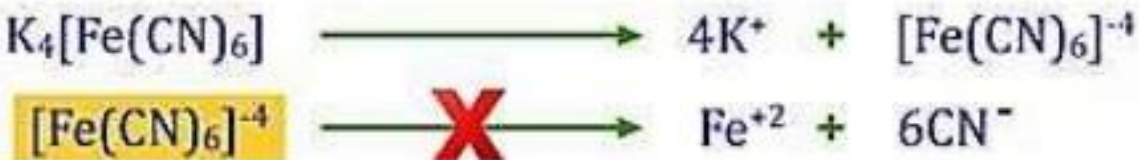
[D] INORGANIC PREPARATIONS

Experiment No.1

Aim

Preparation of Tetramine Copper (II) sulfate monohydrate; $[Cu(NH_3)_4]SO_4.H_2O$.

$$\left[\begin{array}{c} NH_3 \\ \uparrow \\ H_3N :\rightarrow Cu \leftarrow: NH_3 \\ \uparrow \\ \ddot{N}H_3 \end{array} \right] SO_4 H_2O$$
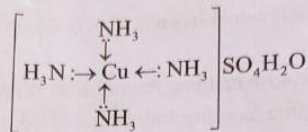
Theory

The name "tetramine copper (II)" indicates that four ammonia molecules are covalently bonded to the copper (II) ion. The formula is written $[Cu(NH_3)_4]^{2+}$. The square brackets signify that the four ammonia molecules and the Cu^{2+} ion act as a group forming a +2 ion. The ion will form part of the solid crystal $[Cu(NH_3)_4]SO_4.H_2O$, in which there is one molecules of water forming a hydrated species.

In aqueous solution of $CuSO_4$, Cu^{2+} ions are bonded to four water molecules in a square planar geometry. The ion is light blue coloured. The water molecules can be displaced by ammonia molecules, which are stronger Lewis bases than water. The appearance of the intense dark blue-violet color of the $[Cu (NH_3)_4]^{2+}$ ion indicates that the reaction has occurred.

$$CuSO_4.H_2O + 4NH_3 \longrightarrow [Cu (NH_3)_4] SO_4.H_2O + 4H_2O$$
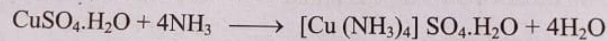
Precaution

Absolute alcohol (ethanol) should be used for this preparation otherwise product does not crystallize out completely.

Chemicals

1) Copper sulfate   3 g
2) Liquor ammonia   6 ml
3) Ethanol   10 ml

Procedure

1) Weigh out approximately 3 g of copper (II) sulfate pentahydrate and record it.
2) Dissolve the copper salt in approximately 12 ml of distilled water in a beaker.
3) Gently heat the above solution to speed up the dissolving process. Then make it cooled upto room temperature by placing beaker in ice-bath or tap water. (Because in hot water tetraammine Copper (II) sulfate get decomposed, do not overheat)

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4) To the cooled solution add approximately 6 ml concentrated ammonia solution slowly with constant stirring.

Initially a bluish white precipitate is formed, further it gets dissolved with forming dark blue solution.

5) To decrease the solubility of the tetra ammine copper (II) complex, add approximately 10 ml of ethyl alcohol with stirring. A deep blue solid precipitates and settles at the bottom.

6) Allow the solid precipitate to stand for 5-10 minutes in ice cold water.

7) Filter the precipitate of the complex on a Buchner funnel and first wash with little amount of cold distilled water, followed by approximately two times 2 ml portions of ethyl alcohol.

8) Allow the product to dry at room temperature on previously weighed watch glass and record the yield.

Observation

Weight of complex Tetramine Copper (II) sulfate monohydrate .... gms

Calculations

1) **Theoretical yield**

$$CuSO_4.5H_2O \quad = \quad Cu(NH_3)_4SO_4.H_2O$$
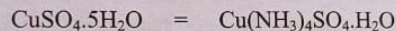
$$249.6 \text{ gms} \quad = \quad 245.6 \text{ gms}$$

$$3 \text{ gms} \quad = \quad 2.952 \text{ gms}$$

2) **Practical yield of complex**

Weight of tetrammine copper (II) sulfate = X = ... gm

3) **Practical % yield**

$$2.952 \text{ gms of complex} \quad = \quad 100\% \text{ yield}$$

$$\therefore X \text{ gms of complex} \quad = \quad \frac{X \times 100}{2.952}$$

$$= \quad ....\% \text{ yield}$$

Result

Sr. No.	Property	Results
01	Practical yield of complex	.... gm
02	% practical yield of complex	.........%
03	Color of the complex	Blue

Cu (29) $\rightarrow$ $1S^2, 2S^2, 2P^6, 3S^2, 3P^6,$ **$4S^2, 3d^9$**

In $CuSO_4$, the oxidation state of Cu is +2. i.e. Cu+2

$Cu(29) = 4S^2 3d^9$

$Cu \rightarrow Cu^{+2} + 2e^-$

$Cu^{+2} = 27$ electrons $= 4S^0 3d^9$

↑↓	↑↓	↑↓	↑↓	↑
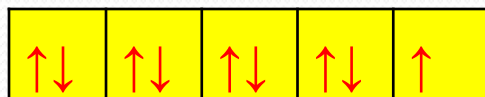
[Cu(NH₃)₄]²⁺ — Oxidation state of Cu²⁺
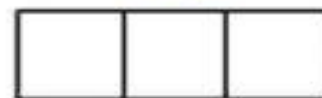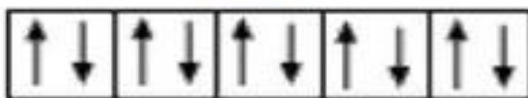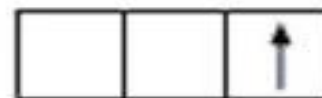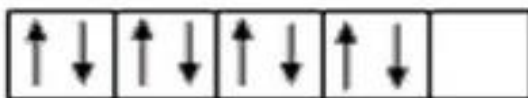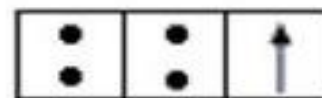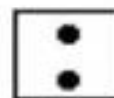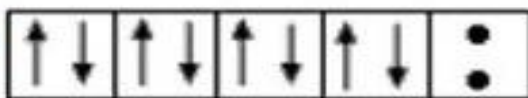
dsp² – square planar, paramagnetic – one unparied electron

Cu^{+2}

3d⁹   4s   4p
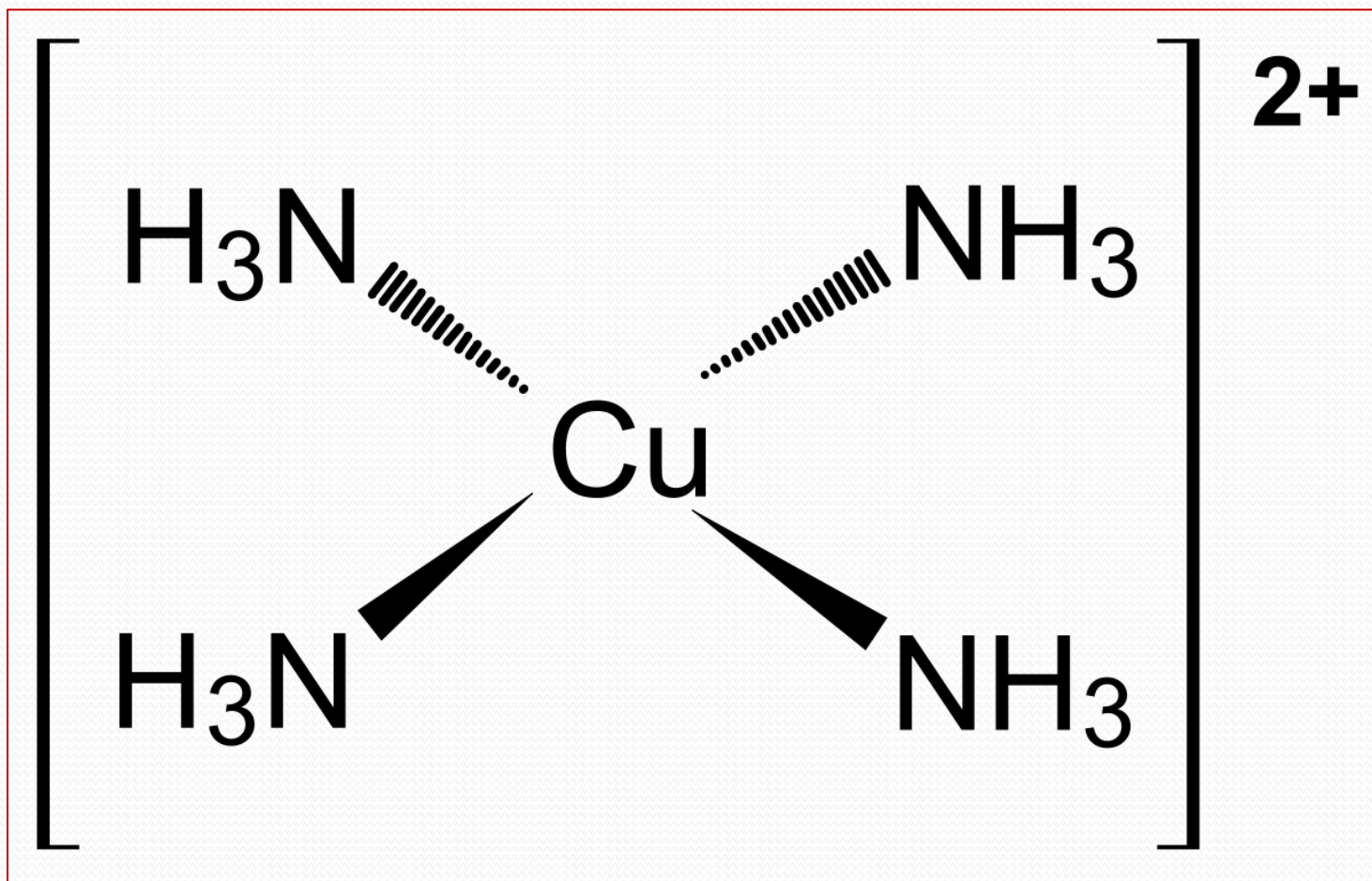
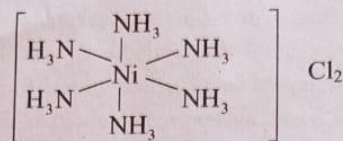Transfer of one electron
from 3d to 4p and
dsp^2 hybridisation

3d   4s   4p

$[Cu(NH_3)_4]^{2+}$

(8 electrons of Cu)

dsp^2 hybrid orbitals
(8 electrons of NH_3
ligands)
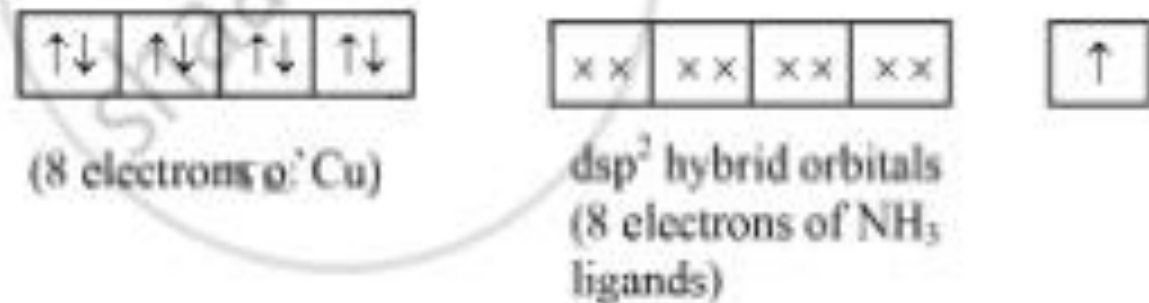
Aim

Preparation of Hexamminenickel (II) chloride, $[Ni(NH_3)_6]Cl_2$.

$$\left[\begin{array}{c} NH_3 \\ H_3N \diagdown \; | \diagup NH_3 \\ Ni \\ H_3N \diagup \; | \diagdown NH_3 \\ NH_3 \end{array} \right] Cl_2$$

Theory

In aqueous solution, nickel ion Ni^{2+} is surrounded by six water molecules which are actually bonded to the central metal ion. This complex is called the hexaquonickel (II) ion.

When ammonia is added to a solution of this salt, ammonia molecules compete with the water in bonding Ni^{2+} and because ammonia forms a stronger bond than water, the ammonia replaces water according to the reaction.

$$NiCl_2 + 6H_2O \longrightarrow [Ni(H_2O)_6]Cl_2$$

$$[Ni(H_2O)_6]Cl_2 + 6NH_3 \longrightarrow [Ni(NH_3)_6]Cl_2 + 6H_2O$$
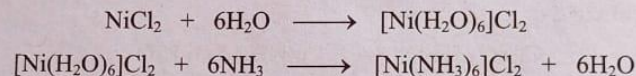
Chemicals

1) Nickel chloride   3 gms
2) Liquor ammonia   10 ml

Procedure

1) Dissolve 3 gms of nickel chloride in 5 ml of warm water in a 100 ml beaker.
2) Add 6 ml of concentrated aqueous ammonia slowly, to a rapidly stirred solution of nickel chloride.
3) Make sure that the color of the solution has changed from pale green to intense violet (if not, add more ammonia)
4) Allow the solution to stand at room temperature for 5 minutes. Cool in an ice-bath, without disturbance, for about 15 minutes.
5) Filter the solution and wash the crystals with concentrated aqueous ammonia solution (~5 ml).
6) Allow the product to dry at room temperature in air, on previously weighed watch glass and record the yield.

Observation

Weight of complex Hexamminenickel (II) chloride .... gms

Calculations

1) Theoretical yield

$$1 \text{ mole of NiCl}_2.6H_2O = 1 \text{ mole of } [Ni(NH_3)_6]Cl_2$$
$$237 \text{ gms} = 231 \text{ gms}$$
$$3 \text{ gms} = 2.9240$$

2) Practical yield of complex

Weight of hexa-amine Ni (II) chloride complex = X = ... gm

3) Practical % yield

$$2.952 \text{ gms of complex} = 100\% \text{ yield}$$
$$\therefore X \text{ gms of complex} = \frac{X \times 100}{2.940}$$
$$= ....\% \text{ yield}$$

Result

Sr. No.	Property	Results
01	Practical yield of complex	.... gms
02	% practical yield of complex	.........%
03	Color of the complex	Pale green

Experiment No. 3

Aim

Preparation of Ferrous Ammonium Sulfate (Mohr's salt) $[FeSO_4(NH_4)_2SO_4.6H_2O]$

Theory

Mohr's salt i.e. ferrous ammonium sulfate $[FeSO_4(NH_4)_2SO_4.6H_2O]$ is a double salt. It can be prepared by making an equimolar solution of hydrated ferrous sulfate and ammonium sulfate in minimum amount of water. A few ml of dil. H_2SO_4 is added to prevent the hydrolysis of $FeSO_4.7H_2O$. Cooling of the hot saturated solution yields light green crystals of Mohr's salt.

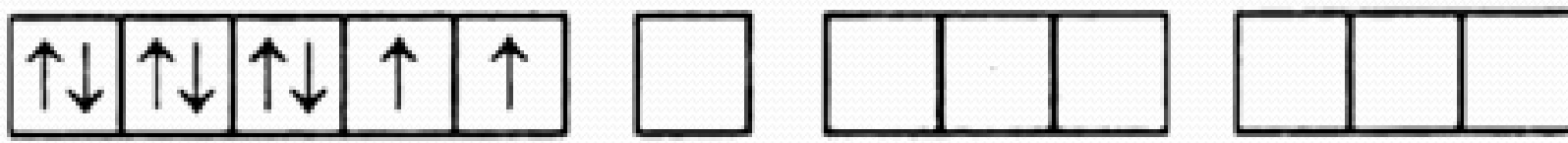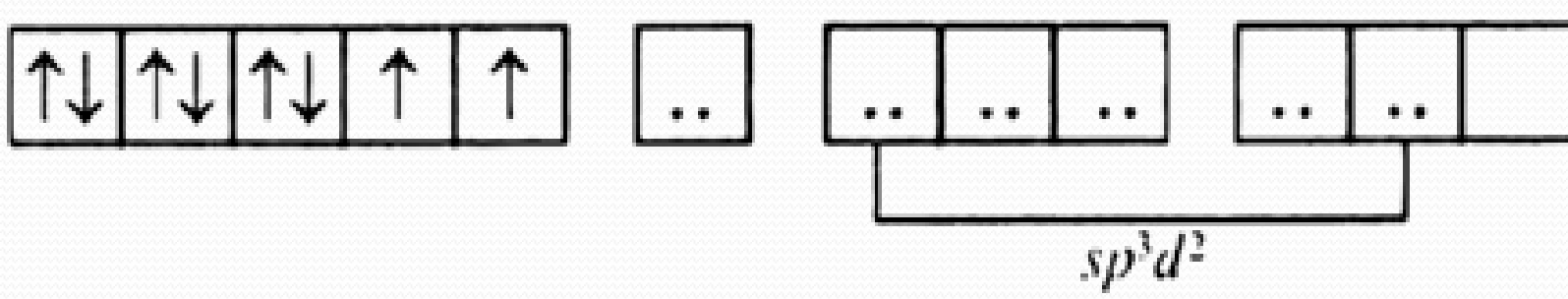$$FeSO_4.7H_2O + (NH_4)_2SO_4 \longrightarrow FeSO_4(NH_4)_2SO_4.6H_2O + H_2O$$

Chemicals

1) Ferrous sulfate — 3 gms
2) Ammonium sulfate — 1.5 gms

3d	4s	4p	4d

Ni(II)

Ni(II) in $[Ni(NH_3)_6]^{2+}$

sp^3d^2

Calculations

1) **Theoretical yield**

$$1 \text{ mole of } NiCl_2.6H_2O = 1 \text{ mole of } [Ni(NH_3)_6]Cl_2$$
$$237 \text{ gms} = 231 \text{ gms}$$
$$3 \text{ gms} = 2.9240$$

2) **Practical yield of complex**

Weight of hexa-amine Ni (II) chloride complex = X = ... gm

3) **Practical % yield**

$$2.952 \text{ gms of complex} = 100\% \text{ yield}$$
$$\therefore X \text{ gms of complex} = \frac{X \times 100}{2.940}$$
$$= ....\% \text{ yield}$$

Result

Sr. No.	Property	Results
01	Practical yield of complex	.... gms
02	% practical yield of complex	.........%
03	Color of the complex	Pale green

Experiment No. 3

Aim

Preparation of Ferrous Ammonium Sulfate (Mohr's salt) [FeSO₄(NH₄)₂SO₄.6H₂O]

$$[FeSO_4(NH_4)_2SO_4.6H_2O]$$

Theory

Mohr's salt i.e. ferrous ammonium sulfate $[FeSO_4(NH_4)_2SO_4.6H_2O]$ is a double salt. It can be prepared by making an equimolar solution of hydrated ferrous sulfate and ammonium sulfate in minimum amount of water. A few ml of dil. H_2SO_4 is added to prevent the hydrolysis of $FeSO_4.7H_2O$. Cooling of the hot saturated solution yields light green crystals of Mohr's salt.

$$FeSO_4.7H_2O + (NH_4)_2SO_4 \longrightarrow FeSO_4(NH_4)_2SO_4.6H_2O + H_2O$$

Chemicals

1) Ferrous sulfate   3 gms
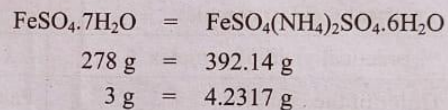2) Ammonium sulfate   1.5 gms

Procedure

1. A mixture of ferrous sulfate (3 gms) and ammonium sulfate (1.5 gms) is dissolved in water (5-20 ml) in a 100 ml beaker containing dilute sulfuric acid (1 ml). If necessary, the mixture is warmed for getting a clear solution.
2. Then the above solution is concentrated by heating beaker on low flame of burner.
3. The solution is concentrated till the crystallization point is reached.
4. Then keep the beaker in ice cold water taken in water bath.
5. After some time (15-20 min), crystals of Mohr's salt separate out from solution.
6. Filter the product on Buchner funnel.
7. Allow the product to dry at room temperature on previously weighed watch glass and record the yield.

Observation

Weight of complex Ferrous Ammonium Sulfate (Mohr's salt) .... gms

Calculations

1) Theoretical yield

$$FeSO_4.7H_2O = FeSO_4(NH_4)_2SO_4.6H_2O$$
$$278 \text{ g} = 392.14 \text{ g}$$
$$3 \text{ g} = 4.2317 \text{ g}$$

2) Practical yield of complex

Weight of ferrous ammonium sulfate, X = ... gms

3) Practical % yield

$$4.2317 \text{ g of complex} = 100\% \text{ yield}$$
$$\therefore X \text{ gms of complex} = \frac{X \times 100}{4.2317}$$
$$= ....\% \text{ yield}$$

Result

Sr. No.	Property	Results
01	Practical yield of complex (X)	.... gms
02	% practical yield of complex	.........%
03	Color of the complex	Faint green