X-ray Crystallographic Studies of Some Heterocyclic Schiff Bases of Salisylaldoxime with Ni (II), Cu (II) and Co (II) Transition Metal Complexes

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Abstract : Four Metal Complexes of the transition metals were prepared by Mixing of metal chlorides [Ni(II), Cu(II) and Co(II)] (0.025M) with salisylaldoxime (0.05 mole) in 1:2 proportion in ethyl alcohol. All the complexes are subjected for XRD analysis. The data obtained from XRD analysis program shows monoclinic crystal system for all the synthesized complexes. From the experimental measurements, various parameters have been estimated. The XRD analysis revealed for all complexes show good intense and sharp peaks, indicating high crystallinity of complexes.

Introduction:

Schiff bases are the most widely used as chelating ligands in coordination chemistry[1]. Schiff base complexes are quite interesting because of the wide variety of possible structures for the ligands, depending upon the different aldehyde and amines used for synthesis. Schiff base metal complexes of the heterocyclic ligands have great industrial and biological applications[2,3]. Most of the Schiff base metal complexes are act as potent metallo drug[4]. Schiff bases are important class of ligand possessing wide range of applications in various fields[5, 6, 7]. Heteronuclear Schiff base complexes have found applications as magnetic materials, catalysts and in field of bio engineering[8]. They are also used in catalysis and in medicine as antibiotics, antiallergic and antitumor agents[9].

Experimental

The salisyladehyde and hydroxylamine were refluxed with each other in ethanol produces salisylaldoxime. Salisylaldoxime were prepared by the standard method[4], in which 20 gm(0.164 mol) of salisyladldehyde in 30 ml alcohol (Rectified sprit) and 15 gm (0.216 mol) of hydroxyl amine hydrochloride in 10 ml water were mixed with each other. Then 10 % solutions of sodium carbonate were added to it so that mixture became alkaline. It was kept for overnight. Then it was acidified with acetic acid and then distilled off the alcohol under reduced pressure later on it was diluted with take twice the volume of water and extracted with 50 ml portion of ether. The ethereal extract were dried with sodium sulphate and allowed the residue to crystallize. Then it was recrystillised from chloroform or light petroleumether (B.P. 40^0C -60^0C). The purity of the product was checked by T.L.C. and by taking M.P. of product (M.P. = 57^0C).

Preparation of simple complexes of Salisylaldoxime

The complexes of Ni-Salisylaldoxime,Cu-Salisylaldoxime and Co-Salisylaldoximehas been synthesized by Mixing of metal chloride [Ni(II), Cu(II) and Co(II)] (0.025M) with salisylaldoxime (0.05 mole). In 1:2 proportion in ethyl alcohol, which gets precipitate by maintaining the P^H with addition of alcoholic ammonia or sodium acetate solution. The corresponding coloured precipitate formed was filtered and washed with hot water and cold methanol. Finally all these metal oxime complexes were dried at 110^0C.

The X-ray powder diffractogram of the metal complexes were used for the structural characterization and determination of lattice dimensions. The observed data of complexes under investigation was compared with other literature data having analogous cell and subsequently indexed to similar geometry.

X-ray Diffraction Study of Cu(II) Complexes

The Cu(II) complexes of ligand PL_1+ SL_1 and PL_2 +SL_2 were subjected to X-ray powder diffraction studies X-ray powder data of all the main peaks have been indexed independently by trial and error method. The data was indexed such that the standard deviation in the lattice parameter values reaches minimum value. The standard deviation observed for Cu(II) complexes is within permissible limit of 2%. The crystal volume is obtained from indexing of the diffraction pattern. The Z value was calculated and rounded up to the nearest whole number. The porosity percentage was calculated from the observed and calculated densities. The density calculated from diffraction data and the observed density was found to be very close to each other indicating perfection in indexing. The observed densities of Cu(II) $(PL_1)_2$ + $(SL_1)_2$ and Cu(II) $(PL_2)_2$ + $(SL_2)_2$ are 1.734, 1.787 gcm^{-3} and calculated densities are 1.814, 1.752 gcm^{-3} respectively. The porosity percentage is 1.143 % and 1.431 % respectively and complex crystallises in the monoclinic crystal system with 2 molecules per unit cell. The probable space group is P2/m.

Conclusively the X-ray powder diffractrograms suggest that the Cu(II) complexes under investigation crystallize with monoclinic crystal system and space group P2/m.

X-ray Diffraction study of Ni(II) Complexes

The complexes of [Ni(II)(PL_2+SL_4)] were used to study the X-ray powder diffrractogram. The crystallographic data and the indexed powder diffraction data is presented in Table 5.26. The standard deviation observed is within the permissible limit. The observed density for [Ni(II)(PL_2+SL_4)] is 1.203 gcm^{-3} while calculated density from Z value and unit cell volume for complexes is 1.2696 gcm^{-3} . The porosity percentage calculated from the observed and calculated densities was found to be 5.322. The crystal system was found to be monoclinic with four molecules per unit cell having probable space group p2/m.

Conclusively, the X-ray powder diffractogram suggest that Ni(II) complexes under investigation crystallize with monoclinic crystal system with probable space group P2/m.
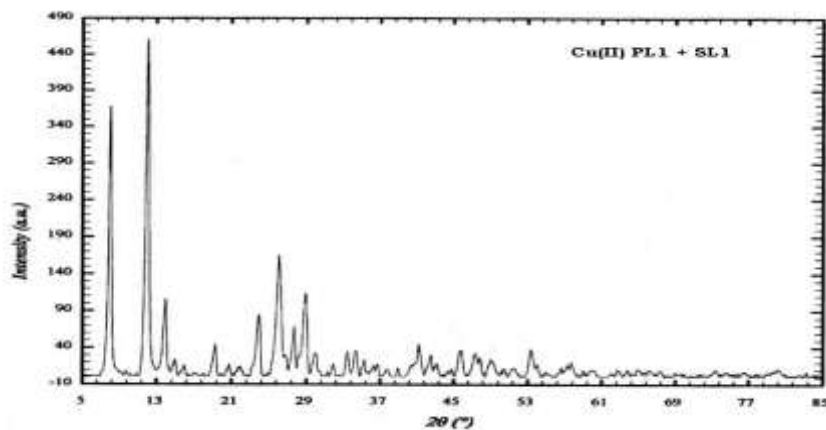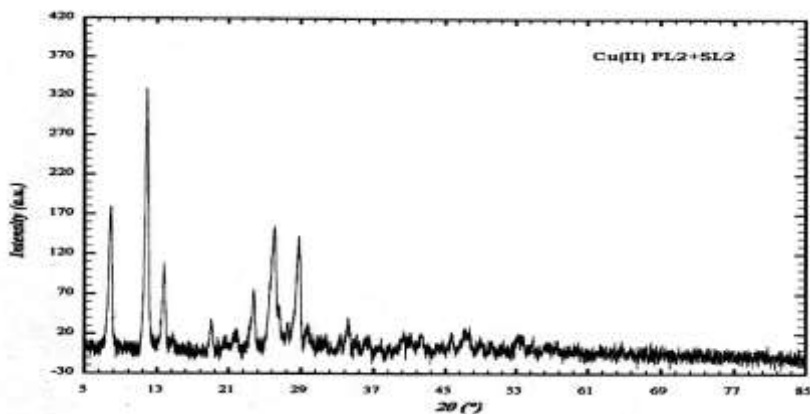
X-ray Diffraction Study of Co(II) Complexes

The X-ray power diffractogram of [Co(II)(PL_1+(SL_3)] complex was selected for the study . The XRD data was fed to computer to get the values of h, k, l, d, for various refluxes using $1/d^2$ values. The standard deviation in crystal parameter found is within permissible limit. The observed density of [Co(II)(PL_1+(SL_3)]is 1.1234 and calculated density is 1.4220. The porosity percentage was calculated from the observed and calculated density which is found to be 21.004 %. The crystallographic data of the complexes fit perfectly in monoclinic crystal system with four molecules per unit cell. The probable spaces group is P2/m.
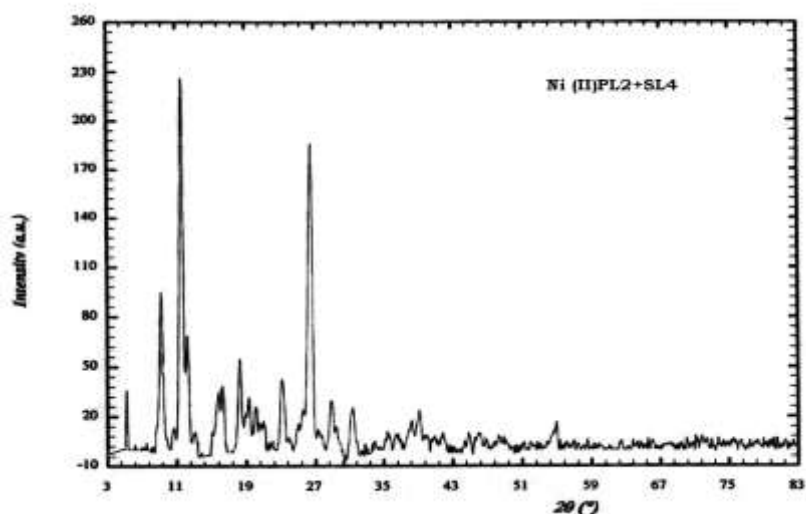
Conclusively the X-ray powder diffractogram suggest that Co(II) complex under investigation crystallize with monoclinic crystal system having space group P2/m .

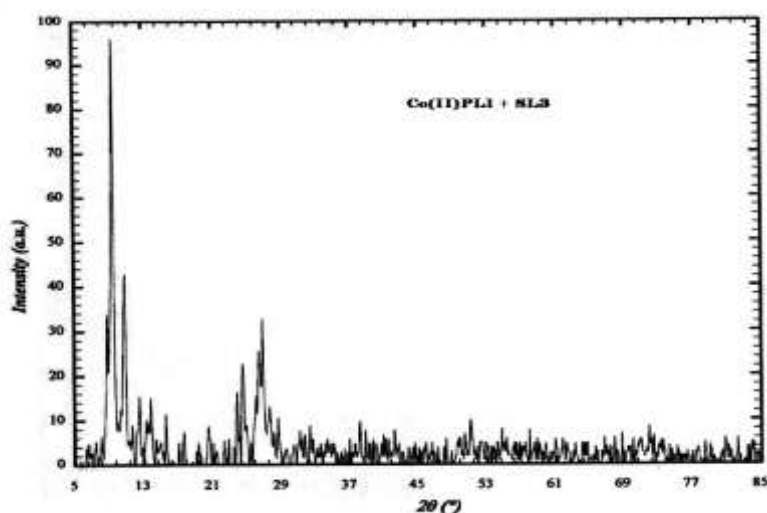**Table-I- X-ray Powder diffraction data of Cu(II), Ni(II) and Co(II) Complexes**

Sr. no.	Compound	D(obs) g/cm^3	D(Cal) g/cm^3	Z	ParticalSize Å	Crystal System	Space Group
1	Cu(PL1+SL1)	1.734507	1.814232	2	246.3716	Monoclinic	P $_{2/m}$
2	Cu(PL2+SL2)	1.787142	1.752233	2	246.3715	Monoclinic	P $_{2/m}$
3	Ni(PL2+SL4)	1.203	1.2696	4	115.656	Monoclinic	P $_{2/m}$
4	Co(PL1+SL3)	1.12346	1.42205	4	246.37	Monoclinic	P $_{2/m}$

X-ray Diffractogram of Cu (II) complex with Ligand PL$_1$+SL$_1$.

X-ray Diffractogram of Cu (II) complex with Ligand PL$_2$+SL$_2$.

X-ray Diffractogram of Ni (II) with Ligand PL$_2$+S

X-ray Diffractogram of Co (II) complex with Ligand PL$_1$+SL$_3$.

Conclusion:

From the above discussion and on the basis of results ofX-ray diffraction, it may be concluded that, the complexes of all metal ions stable transition metal complexes. The XRD analysis revealed that all complexes show good intense and sharp peaks, indicating high crystallinity of complexes.

Acknowledgement

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