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SYNTHESIS AND XRD CHARACTERIZATION OF COPPER (II) CONTAINING METAL COMPLEXES

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Abstract : As part of a continuing search for the synthesis of new antimicrobial agents, some macrocyclic tetradentate nitrogen donor (N4) ligand based transition metal complexes of the type [$ML_{1\text{-}5}$] where M= Cu(II), L_1-L_5 were of our interest. All new metal complexes of transition metal ion containing Cu (II) and ligands [L_1 –L_5] were synthesized. The ligands were prepared by chemical root procedure which included the reaction of pyridin-2-carboxaldehyde with alkyl amines. The XRD data has been obtained by PDXL software. From the experimental measurements, various parameters such as lattice parameter and particle size have been estimated. Particle size is found to be in the range of nanometres. The XRD analysis of the complexes revealed the crystalline nature.

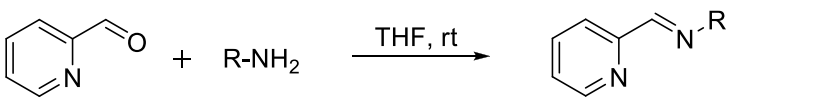
I. INTRODUCTION

Schiff bases derived from an amino and carbonyl compound are an important class of ligands that coordinate to metal ions via azomethine nitrogen and have been studied extensively. In azomethine derivatives, the C≡N linkage is essential for biological activity, several azomethine have been reported to possess remarkable antibacterial, antifungal, anticancer and antimalarial activities Literature survey shows that transition metal complexes generally crystalline as well as amorphous in nature with tetrahedral, octahedral or square planar geometry . In present paper the powder X-ray diffraction of Copper complexes of N-4-disubstituted 1-(pyridine-2yl) methanimine) scanned in the range of 10-60 degree at wavelength 1.540598Å.

The wavelengths are necessary part of crystal system of metal complexes to determine the peak position, miller indices (h, k, l) values, unit cell parameters and 2Θ value with radiation source of CuKα by used as X-ray diffractometer range. The target of X-ray diffraction is to determine the probable structure of the metal complexes. To accomplish this target we must be able to express mathematically the nature of the measured interference pattern in terms of the position of the various atoms within the crystal. To determine the crystalline compounds they are using XRD instrumental technique.

2. Experimental

Necessary chemical reagents were acquired from Sigma-Aldrich and Alfa Aesar, India.
Ligands L1-L5 are synthesized by condensation of pyridine-2-carbaldehyde (475.6 µL, 5.0 mmol), and corresponding amine (1.0 ~ 5.0 equiv.) are dissolved in THF (15 mL) and stirred for 24 h at room temperature.Later, all volatiles are removed under reduced pressure to recover the product.



R= -$CH_2CH_2CH_3$ (L1)
-$CH(CH_3)_2$ (L2)
-$CH_2CH_2CH_2CH_3$ (L3)
-$C(CH_3)_2CH_2CH_3$ (L4)
-$CH_2CH_2CH(CH_3)_2$ (L5)

Fig.1. General procedure for synthesis of ligands L1-L5

Synthesis of N-(pyridin-2-ylmethyl)propan-1-amine (L1).
Ligand L1 is synthesized by following the above general procedure by using pyridine-2-carbaldehyde (475.6 µL, 5.0 mmol) and n-propylamine (1644.2 µL, 20.0 mmol).
Synthesis of N-(pyridin-2-ylmethyl)propan-2-amine (L2).
Ligand L2 is synthesized by following the above general procedure by using pyridine-2-carbaldehyde (475.6 µL, 5.0 mmol) and iso-propylamine (2046.7 µL, 25.0 mmol).
Synthesis of N-(pyridin-2-ylmethyl)butan-1-amine (L3).
Ligand L3 is synthesized by following the above general procedure by using pyridine-2-carbaldehyde (475.6 µL, 5.0 mmol) and n-butylamine (515.8 µL, 5.2 mmol).
Synthesis of 3-methyl-N-(pyridin-2-ylmethyl)butan-1-amine (L4).
Ligand L4 is synthesized by following the above general procedure by using pyridine-2-carbaldehyde (475.6 µL, 5.0 mmol) and iso-pentylamine (603.5 µL, 5.2 mmol).
Synthesis of N-(pyridin-2-ylmethylene)propan-2-amine (L5).
Ligand L5 is synthesized by following the above general procedure by using pyridine-2-carbaldehyde (475.6 µL, 5.0 mmol) and t-pentylamine (1799.5µL,7.5mmol).

3. Procedure for Synthesis of Copper Complexes (CuL1-CuL5)
Complexes CuL1-CuL5 were synthesised by treating N-substituted pyridylimine ligands **(L1-L5)** with the cupric chloride metal salts ($CuCl_2$.6H2O) in methanol (25 mL) under reflux conditions for 24 h. Later, volume was reduced to 1-2 mL and diethyl ether was used for precipitation of complexes. Further, the complexes were air dried and used for characterization

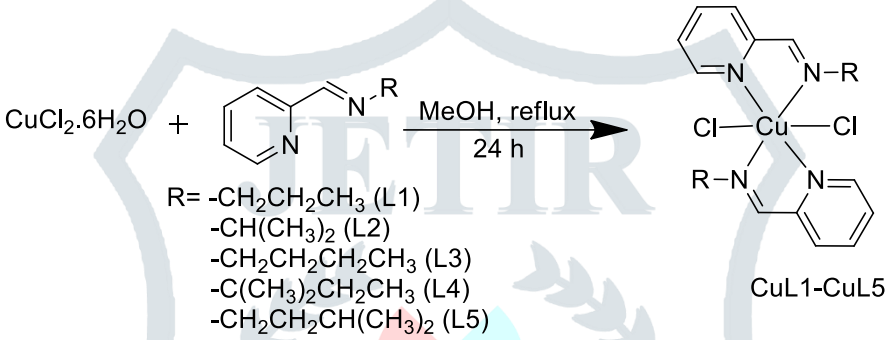


Fig.2.. General procedure for synthesis of complexes CuL1-CuL5.

S.No.	Metal Complexes	Molecular Formula	Abbreviations
1	Bis-(N-Propyl-1-(pyridine-2-yl)methanimine) Copper(II)chloride	$C_{18}H_{24}Cl_2N_4Cu$	CuL1
2	Bis-(N-Isopropyl-1-(pyridine-2-yl)methanimine) Copper(II) chloride	C18H24Cl2N4Cu	CuL2
3	Bis-(N-butyl-1-(pyridine-2-yl)methanimine)Copper(II) chloride	C20H28Cl2N4Cu	CuL3
4.	Bis-(N-tert-pentyl-1-(pyridine-2-yl)methanimine) Copper(II) chloride	C22H32Cl2N4Cu	CuL4
5.	Bis-(N-Isopentyl-1-(pyridine-2-yl)methanimine) Copper(II) chloride	C22H32Cl2N4Cu	CuL5

4. XRD Spectral Analysis of Metal Complex CuL1-Cu L5

The sample is irradiated with a beam of monochromatic x-rays over a variable incident angle range. The X-rays were produced using a sealed tube and the wavelength of the X-ray as 0.154nm (Cu K-alph). The X-rays were detected using a scintillation counting detector. The Interaction with atoms in the sample results in diffracted x-rays when the Bragg equation is satisfied. The X-ray diffraction pattern has been recorded using Brang Banto focusing optic by Rigaku Smartlab diffractometer at Physics Department IIT Indore.

5. Result Analysis & Conclusion

All the samples are characterized at room temperature by X-ray diffraction using Cu Kα radiation. The diffraction pattern of complexes is recorded between two thetas ranging from 10° to 60°. The particle size of the samples is estimated using the Scherrer's formula. According to Scherrer's equation, the particle size is given by $t = 0.9\ \lambda/B\cos\theta$ where t is the crystal thickness (in nm), B is half-width (in radians), θ is the Bragg angle, and λ is the wavelength. The particle size corresponding to each

diffraction maxima are determined from the measurement of the half-width of the diffraction peak. As our synthesized samples are complexes, and due to the lone pair of electrons, distortion occurs due to which it undergoes lower symmetry, yet the characteristic and prominent peaks of complexes and the crystal system have been identified, which are equal and in some cases nearly match the standard diffraction card JCPDS No-01-0765608 & 01-073-6588. The XRD pattern is indicative of their crystalline in nature which is confirmed by the main peaks positioned .The X-ray analysis reveals that the sample is cubic in phase as seen from the presence of extra peaks in XRD pattern. The value of Lattice parameter and the particle size are shown in 'table 1' for all the four complexes. The particle size was found to be within in the range 8.7- 36.2 nm for all the complexes.

S.No.	Metal Complexes	Lattice parameter (Å)	Particle size (nm)
1	CuL1	5.6	36.2
2	CuL2	9.6	31.0
3	CuL3	9.5	30.6
4.	CuL4	6.8	31.5
5.	CuL5	6.1	8.7

Table-1. Lattice parameter and particle size

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