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Synthesis and characterization of copper (II) to Schiff base complexes

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ABSTRACT

Copper (II) Schiff base complexes have been synthesized by treating amines with aldehyde or ketone with diamene in ethanol. All the complexes were characterized on the basis of their micro analytical data, elemental analysis, melting point, IR, UV- Visible Spectra and magnetic moment properties. The magnetic moment suggests that all the complexes are in the range of 1.83 indicating the two unpaired electrons.
The UV- Visible Spectra data shows the $\pi \rightarrow \pi^*$ transition witch suggests two involves molecular orbital π localized at the Azomethane linkage.

Key Words: Schiff bases, Characterization, UV, IR. Molar Conductance

Schiff bases appear to be important intermediate in a number of enzymatic reactions involving interaction of an enzyme with an amino or carbonyl groups of the substrate[1]. One of the most prevalent types of catalytic mechanism in biochemical processes involves condensation of a primary amine in an enzyme, usually that of a lysine residue, with a carbonyl groups of the substrate to form an imine or Schiff base. In fact metal Schiff base complexes have been known since the mid nineteenth century[2] and even before the general preparation of Schiff base ligands themselves[3]. Metal complexes of Schiff bases have occupied a central place in the development of coordination chemistry[4]. A green crystalline product from the reaction of cupric acetate, salicylaldehyde and aqueous ammonia. Schiff bases were used to prepare complexes of metal salicylaldehyde with primary amines[5].

Metal complex of Schiff bases have been extensively studied due to synthetic flexibility, selectivity and sensitivity towards a variety of metal atoms[6]. They are found useful in catalysis, in medicine as antibiotics and anti-inflammatory agents and in the industry as anticorrosion[7-13] agents.

EXPERIMENTAL

The material and reagents used in this study were laboratory pure chemical. The chemical component needed for the synthesis of desired Schiff base complexes were Thionyl chloride, salicylaldehyde, 1, 3-diamino propane, ethylene diamine, p-cresol, 2-isopropyl phenol, 2-tert. butyl phenol, 2-chloro phenol, 2-phenyl Phenol, 2-nitrophenol, glycerol, boric acid, hexamine, ethanol, dichloro methane, Copper acetate etc.

Synthesis of Schiff base Ligands

All the Schiff bases were synthesized by condensing the respective aldehyde with diamine (in 2:1 ratio) in ethanol (rectified). The mixture was refluxed for 3hours on cooling crystals were separated out, which were filtered washed with water and recrystallized from ethanol. The characteristics of all synthesized Schiff bases are given in TABLE-1.

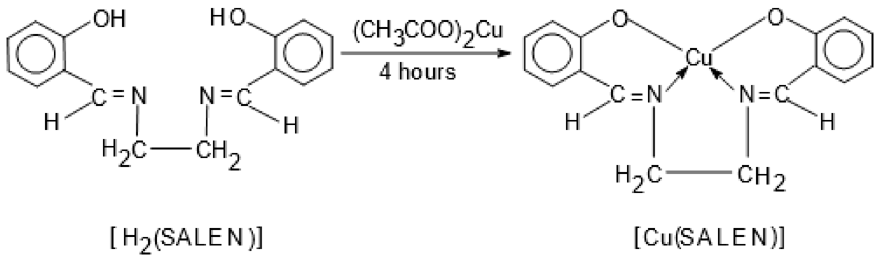
All the Schiff base ligands viz; bis(salicylaldehyde) 1, 2- diaminoethane (H_2SALEN), bis (2-hydroxy-5-methyl benzaldehyde), 1, 2-diaminoethane(H_2HMBEN), bis (2-hydroxy-3-isopropyl benzaldehyde) 1,2- diaminoethane (H_2HPBEN), bis (2-hydroxy-3-phenyl benzaldehyde) 1, 2-diaminoethane(H_2HPHBEN), were synthesized by the procedure as given below.

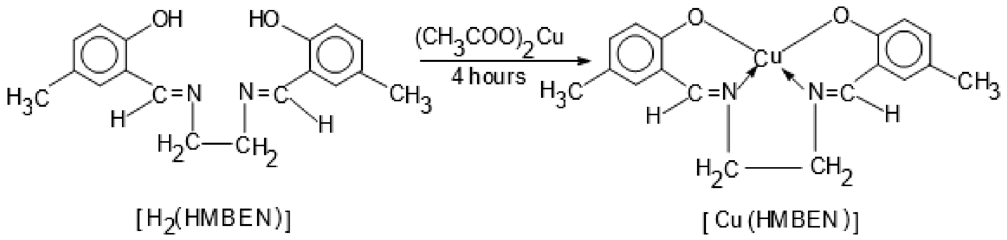
The characteristics of all the synthesized Schiff bases are given in TABLE-1

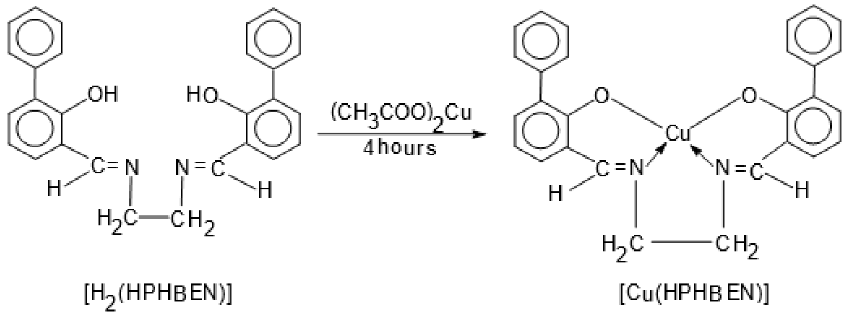
Synthesis of Cu (II) Schiff base complexes

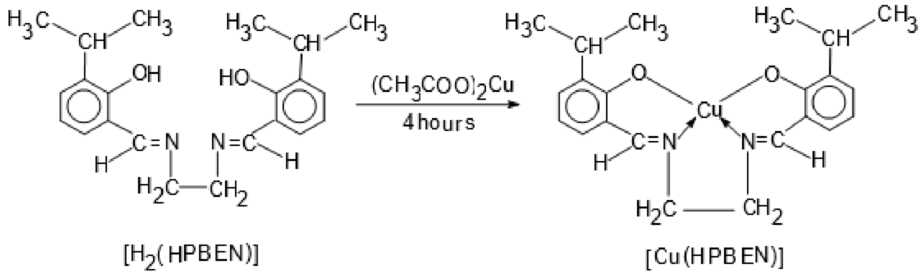
Copper acetate (1.5 m mol) was added into refluxing solution of Schiff bases (1.5m mol) in acetonitrile (12.5c.c). The mixture was refluxed for four hours. On reducing the volume of solvent, complexes got separated out. The precipitate so obtained was filtered washed with a small amount of acetonitrile and dried in vacuum. The purity of complex was checked by thin layer chromatography using dichloromethane: benzene as solvent system.

Similar procedure was adopted for the preparation of other copper (II) complexes.









(TABLE - 1) CHARACTERISTICS OF Cu (II) SCHIFF BASE COMPLEXES

S.NO.	COMPLEX	COLOUR	M.P.*	ANALYTICAL DATA** C %	H %	N %	MOLAR CONDUCTIVITY Ohm^{-1} cm^2 mol^{-1}	MAGNETIC MOMENT μ_{eff} / BM
1.	Cu(SALEN)	Green	280	60.01 (59.07)	4.37 (4.30)	8.89 (8.61)	2.12	1.95
2.	Cu(HMBEN)	Green	240	60.18 (60.16)	5.99 (5.57)	6.22 (6.68)	7.22	1.83
3.	Cu(HPBEN)	Green	210	65.00 (64.54)	6.54 (6.35)	6.21 (5.86)	3.45	2.49
4.	Cu(HPHBEN)	Yellowish Green	272	68.07 (69.85)	4.12 (4.57)	5.78 (5.82)	2.88	2.18

* Decomposition temperature. ** Calculated values are given in parenthesis.

MEASUREMENTS

The Schiff base ligand and its complexes under investigation were subjected to C, H and N Elemental analysis which performed by the micro analytical section of D.R.D.E. Gwalior using CARLO-ERBA and Haereus C, H, N, O-Rapid Elemental Analyser.

For conductance measurement, solutions (10^{-3}M) of metal complexes were prepared in dimethyl formamide (DMF). The conductivity of the solution was measured using a Digital conductivity meter.

Melting point of the ligands and complexes were taken using ''TEMPO'' electrically operated melting point apparatus.

The infrared spectrum of the complexes has been recorded on an FT IR spectrophotometer Magna IR-550 as KBr pallets or in Nujol in the range of 4000-400cm^{-1}.

The electronic spectrum of complexes has been recorded in dimethylformamide using SHIMADZU UV-160A UV-visible spectrophotometer in the range 200-800nm.

Magnetic measurements were carried out at room temperature using a Gouy Balance on a Brucker magnet B-E15.

RESULTS AND DISCUSSION

Chemical Analysis

The complexes were subjected to chemical analysis and result fall within the range expected for the proposed structure. It is seen that experimentally observed analytical values for C, H, and N are in close agreement with the values calculated for the molecular formulae assigned to these complexes.

I.R. Spectral studies

The I.R. spectra in the range of 4000-400cm^{-1} for some of the representative complexes were recorded. The characteristic bands are given in (TABLE-2). There is no absorption band seen in the region 4000-3400cm^{-1} when spectra of copper (SALEN) complex was recorded in Nujol, indicating deprotonation of νOH group and its involvement in coordination. The absorption bands at 3022cm^{-1} and 2922cm^{-1} are assigned to the νC-H stretching. The absorption band in the region 1653-1589cm^{-1} are assigned to the stretching vibrations of unsaturated > C=N– and > C=C < group respectively. The νC–O stretching vibration of hydroxyl group is shifted to 1280–1238cm^{-1}, which suggests that the phenolic hydroxyl group is involved in coordination. An intense band at 669cm^{-1} in the I.R. spectra is due to ν asymm. Stretch of Cu–O band. A band at 740cm^{-1}, which is characteristics of aromatic ortho disubstitution.

The infra red spectra of the Cu (HMBEN) complex shows spectral pattern similar to that observed with Cu (SALEN) complex. In the I.R. spectra of this complex, band in the region 1637–1585cm^{-1} are assigned to the νC=N and νC=C linkages. Strong band at 2912cm^{-1} and 3010cm^{-1} are assigned for the νC–H stretching and absorption band at 1492cm^{-1} is with $-CH_2$ group stretching. A band at 1232cm^{-1} is observed due to νC–O stretching which suggest the involvement of phenolic group in the coordination. Sharp band at 667cm^{-1} and 825cm^{-1} are indicating the presence of Cu–O unit and the phenyl groups respectively.

Cu (HPBEN) complex shows absorption peak at 3373cm^{-1} and 3269cm^{-1} which can be assigned to the various νC-H stretching vibrations of $-CH_3$ and $-CH_2$ group. Strong band at 1602cm^{-1} and 1444cm^{-1} are assigned to the stretching vibration of the unsaturated C=N and C=C groups. The band at 1313cm^{-1} is observed due to νC–O stretching. Which suggest the involvement of phenolic group in the coordination. The absorption band at 692cm^{-1} is indicating the presence of Cu–O unit and band at 819cm^{-1} indicate the aromatic unsymmetric trisubstitution.

In the I.R. spectra of the complex Cu (HPHBEN) peak in the region 3072–3028cm^{-1} can be assigned to the νC-H stretching vibration of $-CH_3$ and $-CH_2$ group. Strong peak at 1583cm^{-1} and 732cm^{-1} are in support of phenyl group. The band at 1606–1583cm^{-1} has been assigned to C=N and C=C linkage. The νC–O shifted to 1269cm^{-1} showing coordination through phenolic hydroxyl group. The peak at 700cm^{-1} can be assigned to Cu–O unit and a peak at 758cm^{-1} supports aromatic vicinal trisubstitution.

Electronic Spectra

The UV–visible electronic spectra of the SALEN ligand showed typical absorptions at 253 and 326nm attributable to ligand $n \rightarrow \pi^*$ and $\pi \rightarrow \pi^*$ charge transfer band, Cu (SALEN) exhibits a broad band centered around 414nm similar to that ascribed in the literature[35, 36].

UV-visible electronic spectra of Cu (HMBEN) complex showed a typical absorption at 415, 530nm and 588nm. An Soret band is obtained at 415nm attributed $n \rightarrow \pi^*$ charge transfer band and another band due to $\pi \rightarrow \pi^*$. The spectrum of Cu (HPHBEN) complex shows very strong band at 599nm which belongs to the charge transfer. The shoulder found in the visible region at 480nm was attributed to the electronic transition ${}^2T_{2(D)} \rightarrow {}^2E_{(D)}$.

Cu (BHBEN) complex obtained at an excitation wavelength of 478nm are shown in. A band in the 565nm and 650nm corresponding to the ${}^4A_{2g} \rightarrow {}^4T_{2g}$ transition in an tetrahedral geometry.

Cu (CHBEN) complex show three band in the 495nm, 572nm and 656nm ranges, representing ${}^4A_{2g} \rightarrow {}^4T_{2g}$, ${}^4A_{2g} \rightarrow {}^4T_{2g}$ and ${}^4A_{2g} \rightarrow {}^4T_{2g}$ transition in the tetrahedral geometry.

ESR Spectra

The ESR spectra of the [Cu (HMBEN)] at 300K shows one intense absorption band in the high field region and is isotropic due to the tumbling motion of the molecules. However, this complex in the frozen state at 77K shows one intense peak in the high field region.

Magnetic Moment

Magnetic moment of copper complexes falls in the 1.83-2.49BM range, which lies within the expected value for one electron.

(TABLE - 2) I. R. FREQUENCY (cm^{-1}) ASSIGNMENTS FOR COPPER(II) SCHIFF BASE COMPLEXES

S.NO.	COMPLEXES	ν C -H	ν C = N	ν C - O	ν Cu - O	ν 
1.	Cu (SALEN)	3022	1653	1280	669	740
2.	Cu (HMBEN)	3010-2912	1637	1232	667	825
3.	Cu (HPBEN)	3373-3269	1602	1313	692	819
4.	Cu (HPHBEN)	3072-3028	1606	1269	700	758

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