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Synthesis and characterization of copper (II) complexes with some Schiff bases

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ABSTRACT

A series of complexes of bi, tri and tetra dentate Schiff bases obtained from 2-hydroxyl-5-methylacetophenone with substituted aromatic amines with metal has been synthesized. The complex have been isolated in solid state and characterized on the basis of elemental analysis, conductivity U.V, I.R. and magnetic susceptibility measurements.

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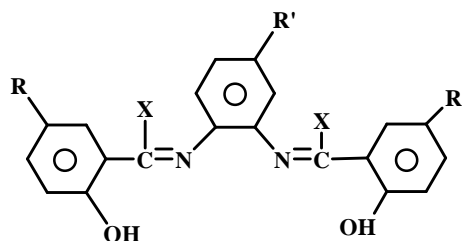
KEYWORDS

Schiff base;
Vibronic;
Spectral evaluation.

INTRODUCTION

The oxygen bridged mono, bidentate, tridentates and tetra dentate complexes have attracted much attention due to their interesting spectral and magnetic properties and their use in bio-chemical processes and homogeneous catalysis^[1-4].

In the present study we have prepared tetra dentate complexes directly by the reaction of Schiff bases derived from 2- hydroxyl-5-methylacetophenone and substituted amines with metals. The complexes obtained are of type [ML] where L is a schiff base molecule as shown below.



where: R=CH₃, R'=CO-C₆H₅, -S-C₆H₅, -S-C₃H₇
X=CH₃, -C₆H₅

EXPERIMENTAL

All the chemicals used were of AR grade (BDHorFLUKA); 2-hydroxy-5-methylacetophenone was prepared by Fries migration reaction of the paracresyl acetate^[5-6]. The Schiff bases were prepared by refluxing 2:1 molar quantities of acetophenone and aromatic amines in ethanol with catalytic quantity of acetic acid for 3 hrs, solidification occurred after concentrating and cooling. The resulting crystals were filtered dried and purified by recrystallisation in ethanol and their purity is checked by elemental analysis, m.p. etc. (TABLE 1). All the schiff bases were yellowish in colour and crystalline in nature.

The complexes were prepared by refluxing ethanolic solutions of the metal chloride and ligands in 1:1 ratio for 2 hrs. After cooling and adjusting the pH to 6.5-7.0 with dilute alcoholic ammonium solution the complex was precipitated out was filtered and washed with hot ethanol and dried in vacuum. The complexes were analyzed for metal ions by standard methods^[7] C, H, N and S were analyzed micro-analytically (TABLE 2).

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RESULTS AND DISCUSSION

All the complexes are very much stable and have high m.p. and are sparingly soluble in common organic solvents. The analytical data of the complexes are recorded in TABLES 2 and 3. The elemental analysis indicates that the metal and ligand is in 1:1 stoichiometry. The conductance measurements were carried out at room temperature using a Thoshniwal conductivity bridge. The electrical conductivity values of 10^{-3} M solutions in DMF (TABLES 2 and 3) were in the range 3.0-12.0ohm⁻¹cm²mole⁻¹ indicating nonelectrolytic nature of the complexes. The electronic spectra of the ligand and complexes were measured by using Shimadzu UV-265 instrument. The ligand and complexes exhibit bands at 265-330 nm due to the vibronic structure of the metal, which seems to be overlapped by the fairly strong ligand to metal charge transfer bands. The intensity of the absorption characteristics of copper (II) ion is much weaker than that of K type of bands. From the experimental data the influence of the metal ion on the

characteristic absorption is much weaker than the ligands which has a large conjugated system^[8].

The IR spectra^[9-10] of the ligands and complexes were recorded in KBr on Shimadzu 435-IR spectrophotometer between 4000- 400cm⁻¹. In the IR spectra of the ligands, bands were obtained in the range 1650-1570cm⁻¹ for ν (C=N) at 1300, 1100cm⁻¹ for ν (C-O) and at about 2900, 2800cm⁻¹ for ν (C-H). The broad weak band in the region 3450-3400cm⁻¹. is due to the intramolecular H-bonded OH. This band totally disappeared in the complexes suggesting the involvement of OH group in coordination. The shift of lower to higher wave numbers of (C=N) or ν (C-O) respectively in the complexes indicates participating of nitrogen atom of the azomethyne group and Phenolic (C-O) in coordination after deprotonation^[11-12]. This was further supported by the disappearance of (OH) stretching vibration in the complexes. The formation of the (M-N) and (M-O) bonds were further supported by the appearance of ν (M-N) and ν (M-O) in the regions 410,430,465cm⁻¹ and 520,540,560cm⁻¹ respectively in the spectra of the complexes.

Magnetic susceptibilities of all the compounds were measured at room temperature (300k) by Gouy balance method. The value of specific susceptibility (χ) and molar susceptibility (χ_M) of all the complexes were determined (TABLE 3). The molar magnetic susceptibilities of the complexes were computed using Pascal's additivity law and the value of susceptibilities for Cu ions required for this purpose were taken from the literature.

The magnetic moments of Copper complexes are in the range 2.04 -2.18 B.M. These values are greater than the spin only values for one unpaired electron (1.73B.M.). The excess value is considered to be due to Spin orbit coupling^[13-14].

From the above studies the tentative structure proposed for the copper complex is as shown in figure below.

TABLE 1 : Analytical data of schiff base ligands

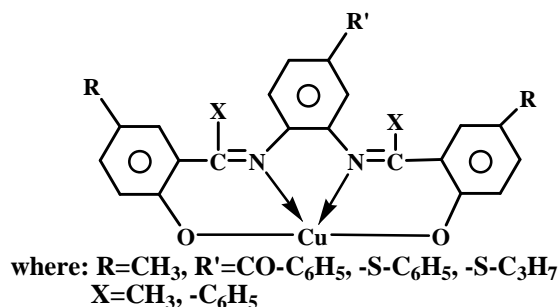
Ligand no.	Ligand (m.f.)	M.W.	M.P °C	% Found (calcd.)			
				C	H	N	S
L ₁	C ₃₁ H ₂₈ O ₃ N ₂	476	210	78.15 (78.18)	5.88 (5.92)	5.88 (5.92)	-
L ₂	C ₃₀ H ₂₈ O ₂ N ₂ S	480	170	75.00 (75.04)	5.83 (5.86)	5.83 (5.86)	6.66 (6.69)
L ₃	C ₂₇ H ₃₀ O ₂ N ₂ S	446	190	72.64 (72.68)	6.72 (6.76)	6.27 (6.30)	7.17 (7.20)
L ₄	C ₄₁ H ₃₂ O ₃ N ₂	600	185	82.00 (82.05)	5.33 (5.38)	4.66 (4.71)	-

TABLE 2: Analytical data of copper (II) complexes

Complex no.	M.W.	Conductivity Ohm ⁻¹ cm ² mol ⁻¹	% Found (calcd.)				
			C	H	N	S	Cu
[Cu L ₁]	537.54	9.1	69.16 (69.20)	4.80 (4.83)	5.15 (5.20)	-	11.79 (11.82)
[Cu L ₂]	541.54	9.3	66.42 (66.47)	4.77 (4.80)	5.14 (5.17)	5.86 (5.90)	11.70 (11.73)
[Cu L ₃]	627.54	9.8	70.71 (70.75)	4.42 (4.47)	4.43 (4.46)	5.06 (5.09)	10.09 (10.12)
[Cu L ₄]	661.54	8.9	74.32 (74.37)	4.51 (4.53)	4.20 (4.23)	-	9.57 (9.60)

TABLE 3: Magnetic susceptibility data of Cu(II) complexes

Complex no.	M.W. (M)	Amount of comp.in gms. (m)	Force of magnetic field (F) in g	χ	χ_M C.G.S	χ_M^{corr} C.G.S	μ_{eff} B.M
[Cu L ₂]	541.54	0.6038	1.8432	2.4666	1335.78	1765.34	2.07
[Cu L ₃]	627.54	0.6512	1.8981	2.3527	1476.44	1858.73	2.12
[Cu L ₄]	661.54	0.7127	1.9289	2.1834	1444.39	1812.86	2.09



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