

NOTE

Synthesis, Characterization and Antibacterial Studies of Co(II), Ni(II) and Cu(II) Schiff Base Complexes derived from Ethyl-methyl Ketone

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Complexes of Co(II), Ni(II) and Cu(II) with Schiff base formed by condensation of 2-aminophenol and ethyl-methyl ketone have been synthesized and their physico-chemical properties have been investigated by microanalytical, UV-Vis, IR spectral, conductance, TGA and magnetic measurements. The ligand field parameters of the complexes have also been calculated. The antibacterial activities have been tested. The complexes are of general formulae $[MLCl_2(H_2O)_2] \cdot 2H_2O$ and $[ML(H_2O)_2] \cdot Cl_2 \cdot 2H_2O$.

Schiff base metal complexes have been a widely studied because of their industrial and biological importance¹. 2-Aminophenol derived compounds show some properties of antimicrobial and pharmacological importance. The present investigation aims at the synthesis, structural and antimicrobial studies of some life-essential 3d-metal ion chelates of ethyl-methyl ketone 2-aminophenol Schiff base.

All the chemicals used were of AR grade.

Synthesis of ligand: The ligand ethyl-methyl ketone-2-aminophenol (Schiff base) was synthesized by adding methanolic solution of ethyl-methyl ketone (4.34 mL) to methanolic solution of 2-aminophenol (5 g) (1 : 1 ratio). The reaction mixture was stirred and then refluxed on water bath for about 5–6 h. The coffee brown condensation product was washed and recrystallised in methanol (m.p. 70°C); (% yield 60%).

Preparation of metal complexes: Metal chelates were synthesized by adding methanolic solution of the appropriate metal salt ($MCl_2 \cdot nH_2O$) to a methanolic solution of Schiff base (in 1 : 1 ratio) and the resulting mixture was refluxed on a water-bath for about 4–5 h. A coloured precipitate appeared on cooling the solution. It was filtered, recrystallised with methanol, washed in petroleum ether and dried under reduced pressure over anhydrous $CaCl_2$ in a desiccator and then in an oven at 80°C. The complexes are air-stable, non-hygroscopic and soluble in DMF and acetone.

Physical measurement: Melting/decomposition point have been determined on a melting point apparatus (Toshniwal, India). Electronic spectra (in MeOH

solution) were recorded on a Perkin-Elmer Spectrophotometer in the region 400–900 nm.

The purity of compounds was monitored by TLC on silica plates. The TGA of only one complex was done at RSIC Nagpur in air (as atmosphere) at a heating rate of 20°C min⁻¹.

TABLE-1
ANALYTICAL AND MAGNETIC DATA OF THE COMPLEXES

Compound Molecular formula [Colour]	m.w. m.p. (dec.) (°C)	Elemental analysis % found (calcd.)				μ_{eff} B.M.	Λ_m ohm ⁻¹ cm ² mol ⁻¹
		C	H	N	M		
C ₁₀ H ₁₃ NO (ligand) [Coffee brown]	163.00 (70.1)	73.0 (73.6)	7.80 (7.97)	8.48 (8.58)	—	—	—
[Co(C ₁₀ H ₁₃ NO)Cl ₂ (H ₂ O) ₂].2H ₂ O [Dark brown]	364.90 (215)	32.56 (32.88)	5.68 (5.75)	3.78 (3.83)	16.10 (16.08)	5.02	25.00
[Ni(C ₁₀ H ₁₃ NO)Cl ₂ (H ₂ O) ₂].2H ₂ O [Dark brown]	365.70 (210)	32.16 (32.90)	5.67 (5.75)	3.76 (3.83)	16.12 (16.09)	2.97	17.00
[Cu(C ₁₀ H ₁₃ NO)(H ₂ O) ₂].Cl ₂ .2H ₂ O [Blackish brown]	369.50 (220)	32.48 (32.47)	5.63 (5.68)	3.67 (3.78)	17.05 (17.18)	1.89	112.00

The analytical data of the complexes show that the metal to ligand stoichiometry is 1 : 1. The conductivity data (in methanol, 10⁻³ M) suggest non-electrolytic nature of the Co²⁺ and Ni²⁺ complexes while the Cu²⁺ complex shows 1 : 2 electrolytic nature.

The IR spectrum of the ligand shows a strong band at 1588 cm⁻¹ due to $\nu(\text{C}=\text{N})$ group. On chelation this azomethine group shifts to lower frequency side (1580 cm⁻¹) indicating the participation of this group in chelation.^{2,3} The characteristic phenolic band which appeared at 1389 cm⁻¹ $\nu(\text{O}-\text{H})$ and 1272 cm⁻¹ $\nu(\text{C}-\text{O})$ in the spectra of ligand (base) shifts at 1405 cm⁻¹ and 1305 cm⁻¹ in the complexes. This suggests participation of phenolic group in complexation. The presence of water molecules within coordination sphere is supported by the appearance of bands at 3436 ± 20 cm⁻¹ (br) and 760 ± 25 cm⁻¹ (sharp), attributable to stretching and rocking mode respectively. The new bands at 550–500 cm⁻¹ and 450–400 cm⁻¹ in the corresponding metal complexes have tentatively been assigned to $\nu(\text{M}-\text{N})$ respectively.

The electronic spectra of the Co(II) complex shows two bands at 17869 cm⁻¹ (ν_2) and 19546 cm⁻¹ (ν_3). The values of 10 Dq, B, β , ν_2/ν_1 and LFSE are 10160 cm⁻¹, 952 cm⁻¹, 85, 2.31, 114.6 kJ mol⁻¹ respectively. This is in fair agreement with the literature values for octahedral geometry.^{5,6} The Ni(II) complex shows three bands at 11123 cm⁻¹ (ν_1), 17211 cm⁻¹ (ν_2) and 23584 cm⁻¹ (ν_3). The values of 10 Dq, B, β , ν_2/ν_1 and LFSE are 11123 cm⁻¹, 495 cm⁻¹, 0.45, 1.54, 160.7 kJ mol⁻¹ respectively, suggesting an octahedral geometry. The Cu(II) complexes gives two bands at 12121 cm⁻¹ and 18115 cm⁻¹, suggesting the square-planar geometry. The observed magnetic

moment values for the Co(II), Ni(II) and Cu(II) complexes are as 5.02, 2.97 and 1.89 B.M. respectively.⁷

Thermogram of copper complex shows a loss in weight corresponding to two lattice water molecules, which occurs up to 210°C (% wt. loss 9.00/9.74). On increase of temperature upto 290°C, a further loss in weight has been observed. This corresponds to two molecules of coordinated water (% wt. loss 17.00/19.48). The thermal degradation of the dehydrated metal chelate begins on heating above 290°C, and the pyrolysis curves exhibit this process to complete roughly in two steps. A constant weight thermal curve has been observed after 450°C. The remaining percentage weight in this horizon is 20.00/19.35 which corresponds to metal oxide.

Antibacterial activity: All the three compounds and base (ligand) have been evaluated for their antibacterial activities against *Bacillus Subtilis*, *Salmonella typhi* and *Escherichia coli* at concentration 25 and 50 µg/mL by agar growth technique.⁸ Cu²⁺-complex shows more activity than Co(II) and Ni(II) complex (Cu > Co ≈ Ni). Metal complexes exhibited more activity than the Schiff base alone.

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(Received: 11 December 1997; Accepted: 17 February 1998) AJC-1460