

NOTE

Synthesis and Structural Studies of Co(II), Ni(II), Zn(II) and Cd(II) Metal Complexes of 2-Hydroxy-5-Methyl Benzene-1,3-bis-(Carbalidine-2-Amino Thiazole)

R.C. SHARMA*, SHUBHRA SINGH, D. KUMAR and RITIKA VATS
*Department of Chemistry, Dr. B.R. Ambedkar University, Institute of Basic Sciences
Khandari Road, Agra-282 002, India*

Some new metal complexes of Co(II), Ni(II), Zn(II) and Cd(II) of 2-hydroxy-5-methyl benzene-1,3-bis-(carbalidine-2-amino thiazole) have been synthesized. All complexes and ligands are characterized on the basis of their repeated melting point determination, TLC, elemental analysis, conductance measurements, magnetic susceptibility measurement, UV and IR spectra data.

Key words: Synthesis, structural, Co(II), Ni(II), Zn(II) and Cd(II) Schiff base, complexes

Schiff base metal complexes have been widely studied because of their vast industrial and biological applications.¹ They are also found to have a number of pharmacological utility.² Schiff base ligands obtained from condensation of 2-hydroxy-5-methyl benzene-1, 3-dicarbaldehyde with amino compounds are found to be good carriers for specific transport of some metal ions.³

In continuation of our earlier work⁴, we have prepared and characterized the ligand 2-hydroxy-5-methyl benzene-1,3-bis-(carbalidine-2-amino thiazole) and its metal complexes with Co(II), Ni(II), Zn(II) and Cd(II).

All chemicals used were of AR grade. 2-Hydroxy-5-methyl benzene-1,3-dicarbaldehyde was prepared according to the procedure given by Gange *et al.*⁵

Preparation of Ligand: Schiff base ligand 2-hydroxy-5-methyl benzene-1,3-bis-(carbalidine-2-amino thiazole) (HMBCAT) was synthesized by addition of the methanolic solution of synthesized dicarbaldehyde (2-hydroxy-5-methyl benzene-1,3-dicarbaldehyde) (HMBD) and 2-amino thiazole in 1 : 2 molar ratio and refluxing it for 8–10 h. On cooling yellowish rust crystalline solid was isolated which was washed with alcohol, ether, recrystallized from methanol and dried.

Preparation of Complexes: A solution of metal salts in water and a methanolic solution of ligand HMBCAT in 1 : 2 molar ratio were mixed and refluxed for 3 h. When the refluxed solution was cooled, coloured solids separated out. The resulting complexes were filtered, washed with alcohol, ether and dried in vacuum.

Carbon, hydrogen and nitrogen analyses were carried out on Carlo-Erba Micro

Analyser (Model 1106) and sulphur was estimated as BaSO₄ by standard procedure.⁶ Nickel and cobalt were estimated by precipitating them as their pyridine complexes.⁷ IR spectra were recorded on a JASCO spectrophotometer-0087 in KBr medium. The conductance measurements were carried out on Toshniwal conductivity bridge using 10⁻³ M DMF solutions. The UV spectra were recorded on Shimadzu digital double beam spectrophotometer (Model UV150-150.02). The magnetic susceptibility was determined at room temperature by Gouy's method.

The analytical data (Table-1) of complexes reveal their 1:2 (ML₂) stoichiometry and the conductivity measurements suggest their non-electrolytic nature.

In IR spectra of dicarbaldehyde bands in the region of 1715–1695 cm⁻¹ and at 2880 cm⁻¹ were obtained due to the stretching vibration of >C=O and —C—H (aldehyde) respectively. The bands due to phenolic group and CH—aromatic were obtained at 3525 cm⁻¹ and 3000 cm⁻¹ respectively.

In the IR spectra of ligand (HMBCAT) some bands in the region of 1640–1630 cm⁻¹, 1270–1190 cm⁻¹, 1310–1220 cm⁻¹ and 3550–3400 cm⁻¹ were obtained. These are due to stretching vibration of azomethine group, (C—S) bond, (C—S—C) bond and phenolic group respectively.

TABLE-1
PHYSICAL AND ANALYTICAL DATA OF DIALDEHYDE, SCHIFF BASE AND METAL CHELATES

Compound	m.f.	m.p.	% Analysis, found (calculated)				
			C	H	N	S	M
HMBD (Yellow)	C ₉ H ₈ O ₃	130	65.76 (65.85)	4.66 (4.88)	—	—	—
HMBCAT (Shiny yellowish rust)	C ₁₅ H ₁₂ N ₄ OS ₂	110	54.79 (54.87)	3.49 (3.65)	17.68 (17.07)	19.23 (19.51)	—
[Co(HMBCAT) ₂] (Yellowish brown)	[(C ₃₀ H ₂₂ N ₈ O ₂ S ₄)Co]	250	50.33 (50.49)	3.38 (3.09)	15.34 (15.71)	17.25 (17.95)	7.86 (8.26)
[Ni(HMBCAT) ₂] (Turmeric yellow)	[(C ₃₀ H ₂₂ N ₈ O ₂ S ₄)Ni]	232	50.34 (50.51)	3.41 (3.09)	15.35 (15.71)	17.24 (17.96)	7.89 (8.24)
[Zn(HMBCAT) ₂] (Yellow)	[(C ₃₀ H ₂₂ N ₈ O ₂ S ₄)Zn]	220	50.13 (50.24)	3.48 (3.06)	15.33 (15.57)	17.25 (17.79)	8.87 (9.09)
[Cd(HMBCAT) ₂] (Yellowish rust)	[(C ₃₀ H ₂₂ N ₈ O ₂ S ₄)Cd]	230	46.83 (46.97)	2.58 (2.87)	14.23 (14.61)	16.37 (16.70)	13.87 (14.66)

The IR spectra of metal complexes show this shifting of bands towards the negative region by 20–30 cm⁻¹ suggesting the coordination of ligand to metal. The band due to phenolic group is absent in the complexes indicating the

deprotonation of phenolic O—H group. The band due to azomethine group is also shifted to the lower region, which reveals the coordination of ligand to metal through nitrogen. However, no shift in the band position due to $>C=N$ (cyclic) around $1490\text{--}1475\text{ cm}^{-1}$ is observed which infers that the nitrogen of the thiazole ring of the ligand does not coordinate.

In the electronic spectra of Co(II) complexes, three bands were obtained in the region of $15820\text{--}13240\text{ cm}^{-1}$ and $19850\text{--}17950\text{ cm}^{-1}$, which correspond to the transitions ${}^4T_{1g}(F) \rightarrow {}^4T_{2g}(v_1)$ and ${}^4T_{1g}(F) \rightarrow {}^4T_{1g}(P)(v_2)$, respectively. These transitions suggest an octahedral geometry around the metal ion. The magnetic moment values (3.7 B.M.) of the complexes support the octahedral geometry. The appearance of three bands in the electronic spectra of Ni(II) complexes in the region of $11330\text{--}10220\text{ cm}^{-1}$, $18540\text{--}16300\text{ cm}^{-1}$ and $26540\text{--}25420\text{ cm}^{-1}$, correspond to the transitions ${}^3A_{2g} \rightarrow {}^3T_{1g}$, ${}^3A_{2g} \rightarrow {}^3T_{1g}F(v_2)$ and ${}^3A_{2g} \rightarrow {}^3T_{1g}P(v_3)$, respectively also suggests an octahedral geometry around the central metal ion. Its magnetic moment values (2.83 B.M.) support the octahedral geometry.

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Contact:

Prof. Antje Baeumer

E-mail: ajb23@cornell.edu