Available online at www.derpharmachemica.com

ISSN 0975-413X
CODEN (USA): PCHHAX

Der Pharma Chemica, 2017, 9(17):7-9
(http://www.derpharmachemica.com/archive.html)

Interaction of Schiff Base Derived from 4-chloro-5-sulfamoyl-2',6'-salicyloxylidide with Ni(II), Co(II) and Cu(II)

Suparna Ghosh[1*], Shweta Sharma[1], Ruchi Dubey Sharma[1], Anita K[1], Suman Malik[2], Bharti Jain[3]

[1]*Department of Chemistry, Career College, Bhopal-462023, India*
[2]*Department of Chemistry, Sadhu Vaswani College, Bairagarh, Bhopal, India*
[3]*Department of Chemistry, Govt. SNGPG (Auto) College, Bhopal, India*

ABSTRACT

A new unsymmetrical bidentate Schiff base was synthesized using 4-chloro-5-sulfamoyl-2',6'-salicyloxylidide (xipamide) and salicylaldehyde. The Ni(II), Co(II) and Cu(II) complexes of this Schiff base of ML_2 type have been synthesized and characterized by elemental analysis, conductivity, magnetic measurements, IR and electronic spectra studies. The conductivity data of the complexes suggests their non-electrolytic nature. The antibacterial activities of the ligand and its complexes are also studied. The antibacterial experiments indicate that the ligand and its complexes possess antibacterial activity against Escherichia coli and Bacillus subtilis and that the complexes have higher activity than that of the Schiff base.

Keywords: Schiff base, Xipamide, Elemental analysis, Antibacterial activity, Conductivity

---

INTRODUCTION

Schiff bases are an important class of ligands in coordination chemistry and widely used in different fields [1]. Unsymmetric complexes are very important because of their medicinal properties [2,3]. To continue the investigation in this area, a new unsymmetrical Schiff base (Figure 1) synthesized from 4-chloro-5-sulfamoyl-2',6'-salicyloxylidide (xipamide) and Salicylaldehyde. The Schiff base synthesizes in this paper is a chelating agent and the chelation of it with metal ion may increase it biocidal effect [4,5]. In the present communication, we report the synthesis, characterization and antibacterial activities of Schiff base ligand and its complexes with Fe(II) and Co(II). This paper offers a new significance in the field of coordination chemistry.

MATERIALS AND METHODS

All the chemicals used in present work were of AR grade (anhydrous) and purchased from Merck (USA). Pure sample of Xipamide (XM), molecular formula $C_{15}H_{15}ClN_2O_4S$, molecular weight 354.81 was obtained from Dishman pharmaceuticals, Gujrat.

Preparation of Schiff base

Equimolar (0.01 M) solutions of pure drug and salicyladehyde were separately prepared in methanol-water mixture (1:1) and refluxed for 5 h and kept for two days. Peach colour crystals of xipamide-salicylaldimine (XM-SA) Schiff base were formed in the reaction mixture, which were filtered and washed thoroughly with 50% methanol, dried over vacuum and weighed. Melting point and yield of Schiff base was reported in Table 1.

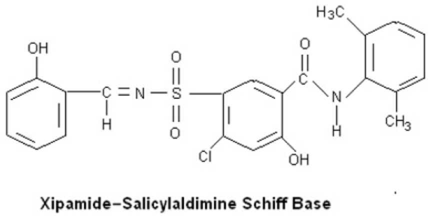

Figure 1: Structure of ligand

Synthesis of complexes

For the synthesis of complexes ligand-metal ratio was confirmed by conductometric titrations using monovariation method on systronics conductivity meter using dip type electrode. Conductometric titrations supported 2:1 (L:M) ratio in the complex which was further supported by Job's method [6] of continuous variation as modified by Turner and Anderson [7]. The stability constant and free energy change values were also calculated.

The metal complexes were prepared by refluxing 60% acetone solution of ligand (0.006 M) and metal salt (0.003 M) for 4 h. The refluxed solutions were kept for 5-6 days. Solid crystalline compounds appeared in the solution, which were filtered, washed with 60% acetone and dried over fused $CaCl_2$.

Analytical procedure

Elemental analyses were carried out on a model 2400 Perkin elemental analyzer from CDRI Lucknow. Metal contents were determined gravimetrically. The infrared spectra were measured on a Nicolet 400 D FTIR spectrophotometer using KBr pellets from Vikram University, Ujjain. The electronic spectra of the metal complexes in Dimethyl Formamide (DMF) were recorded on JASCO 7800 Elico SL-159 and Shimadzu UV-160A UV-Visible spectrophotometer from Vikram University, Ujjain. The X-ray Diffraction (XRD) studies were carried at SICART, Gujrat using Cu target X-ray tube. Magnetic susceptibility measurements of the complexes in the solid state were determined by Gouy balance using $CuSO_4$ as the calibrant at room temperature from Centre for Advance Technology, Indore. Molar conductance measurements were made in anhydrous DMF on a Systronic model 305 conductivity bridge. The melting points of the ligand and complexes were recorded in open capillaries on a capillary melting point apparatus.

Antibacterial activity

Above synthesized compounds and ligands (Schiff base) were screened against bacteria *Escherichia coli* and *Bacillus subtilis* using Streptomycin as standard by the filter paper disc method [8] at various concentrations using nutrient agar as medium. Sterilized filter paper of 5 mm diameter were soaked in solutions of different concentrations of test samples and introduced on nutrient agar plates. These plates were incubated for 48 h at 35°C.

RESULTS AND DISCUSSION

On the basis of physicochemical characteristics it has been found that the complexes are non- hygroscopic, stable at room temperature, insoluble in water but fairly soluble in Dimethyl Sulfoxide (DMSO). The magnetic moment data indicates that the complexes are paramagnetic in nature. The molar conductance values for all the complexes in 10^{-3} DMSO are in the range of 9.5-14 $\Omega^{-1}.cm^2.mol^{-1}$ suggesting their non-electrolytic nature [9]. Elemental analysis data, formula weights and melting points are given in Table 1.

Table 1: Physicochemical and analytical data of ligand and complexes

Ligand/Complex	Elemental analysis % found (calculated)						Melting point (°C)	Color	Yield (%)	μ_{eff} (BM)
	C	H	N	S	Cl	M				
$C_{22}H_{19}N_2O_5ClS$	56.87	4.04	5.84	6.91	7.5	----	250	Peach	57.3	--------
	-57.77	(4.14)	(6.10)	-6.97	-7.74					
$(C_{22}H_{19}N_2O_5ClS)_2Ni.2H_2O$	52.01	3.23	5.34	6.39	7.05	5.75	230	Light green	37	2.94
	-52.29	-3.56	-5.54	-6.33	-7.03	-5.81				
$(C_{22}H_{19}N_2O_5ClS)_2Co.2H_2O$	51.98	3.68	5.45	6.03	7.01	5.79	232	Bluish green	43	4.63
	-52.29	-3.56	-5.54	-6.33	-7.03	-5.83				
$(C_{22}H_{19}N_2O_5ClS)_2Cu.2H_2O$	52.07	3.94	5.52	6.31	7	6.26	238	Dark green	57	1.83
	-52.27	-3.12	-5.12	-6.11	-6.98	(6.1 3)				

Electronic spectra and magnetic measurements

The electronic spectral measurements were used for assigning the stereochemistry of the metal ions in the complexes based on position and number of d-d transition peaks. The electronic spectrum of Ni(II) complex shows three bands at 10242 cm^{-1}, 16925 cm^{-1} and 25350 cm^{-1} assigned to spin allowed transitions ${}^3A_{2g}(F) \rightarrow {}^4T_{2g}$ (F), ${}^3A_{2g}(F) \rightarrow {}^4T_{1g}$ (F) and ${}^3A_{2g}(F) \rightarrow {}^4T_{1g}$ (P) [10]. Similar types of transitions are reported for octahedral Ni (II) complexes. The magnetic moment value of Ni (II) complex is 2.94 B.M. indicating an octahedral geometry [11] of this complex. Electronic spectrum of Co(II) exhibits two bands at 18230 and 22510 cm^{-1} which may be assigned to the transitions ${}^4T_{1g}(F) \rightarrow {}^4A_{2g}$ (F) and ${}^4T_{1g}(F) \rightarrow {}^4T_{1g}$ (P) transitions, considering an octahedral geometry [12] also supported by its magnetic moment value (4.63 B.M) [13]. The electronic spectra of Cu(II) complex shows two energy bands at 33070 cm^{-1} and 25870 cm^{-1} due to ${}^4T_{2g}(F) \rightarrow {}^4A_{2g}$ (F) and ${}^4T_{1g}(F) \rightarrow {}^4T_{2g}$ (P) transitions respectively indicating an octahedral geometry around Cu(II) [14].

Infrared spectra

The IR spectra of Schiff base shows a sharp band near 1639 cm^{-1} which may be due to azomethine linkage and shows lowering in frequency in metal complexes indicating the coordination of metal ions through azomethine nitrogen [15]. The ligand shows strong band at 3386 cm^{-1} due to phenolic–OH group [16]. This band is absent in all the metal complexes indicating the involvement of this group in complex formation [17]. Moreover, the shift of the $\nu_{C\text{-}O}$ phenolic bands from 1282 cm^{-1} in ligand to 1282-1327 cm^{-1} in the spectra of metal complexes supports the coordination of the phenolic oxygen atom to the metal ion [18]. Bands observed at 1163 ± 5 cm^{-1} are characteristics of SO_2-N linkages in Schiff base and complexes respectively [19]. The M-N bands [20] appeared in the range of 570-580 cm^{-1} and bands for M-O modes [21] appeared in the range of 505-521 cm^{-1} in all the complexes indicates that Schiff base is coordinated through O and N atom [22]. Absorption bands at 3500 cm^{-1} and 3504 cm^{-1} show the presence of water of coordination in complexes [23].

Hence on the basis of elemental analysis, magnetic moment data, conductivity measurements and spectral studies the geometry of the complexes can be presumed to have octahedral geometry as shown in Figure 2.

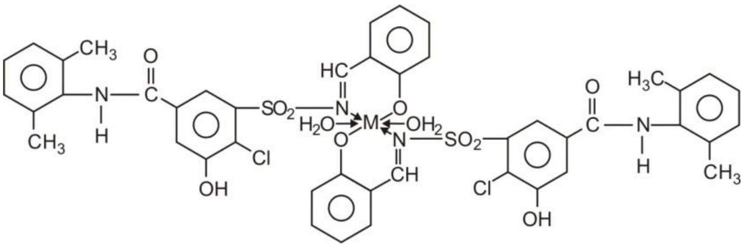

Figure 2: Octahedral geometry
(M=Ni/Co/Cu)

Antibacterial activity

Antibacterial activity of ligand and their metal complexes are given in Table 2. The zone of inhibition based upon size around the disc was measured. Antibacterial activity of the ligand and complexes reveal that the activity zones of inhibition for the complexes against *Escherichia coli* except is higher as compared to ligand. Co(II) complex of the ligand showed higher activity with 25 mm inhibition whereas the standard drug streptomycin showed 18 mm inhibition at the same concentration.

The antibacterial activity of all the complexes against *Bacillus subtilis* showed higher activity with 12-23 mm inhibition. Standard drug streptomycin showed only 16 mm inhibition at the same concentration as of the test drug. On the basis of these observations it can be said that complexation or chelation increases the antibacterial activity [24,25].

Table 2: Antibacterial screening data of the ligand L and its complexes

S. No.	Ligand/complexes	Antibacterial activity zone of inhibition (in mm)	
		Escherichia coli	*Bacillus subtilis*
1	L	10.22	11.11
2	$(CoL_2(H_2O)_2)$	25.28	23.44
3	$(NiL_2(H_2O)_2)$	16.11	12.30
4	$(CuL_2(H_2O)_2)$	15.23	13.46
5	Streptomycin (Standard)	18.00	16.23

ACKNOWLEDGEMENTS

The author’s gratefully acknowledge the Principal and management of Career College for providing proper laboratory facilities. We also pay heartfelt thanks to Dr. Suman Malik, Sadhu Vaswani College, Bairagarh, Bhopal and Dr. Bharti Jain, Sarojini Naidu Autonomous Girls College, Bhopal for their guidance and motivation. Authors are also indebted to CDRI, Lucknow for providing the facilities of elemental analysis and Vikram University Ujjain for recording IR and electronic spectra.

REFERENCES

[1] B.C. Yang, J.F. Yuhug, B. Shuangyu, Z. Dongmei, Z. Xia, *Asian. J. Chem.*, **2011**, 23(12), 5477.
[2] W. George, D.M.G. Okel, *Inorg. Chim. Acta*., **1984**, 82, 101.
[3] B.K. Singh, B. Narender, Prakash Anant, *Eur. J. Chem.*, **2012**, 9(2), 532.
[4] M.B. Ferrari, C.S. Bisceglie, F.G. Pelosi, P. Tarasconi, *Inorg. Chim. Acta*., **2001**, 312, 81.
[5] K.D. Rainsford, M.J. Whitehouse, *Pharm. Pharmacol*., **1976**, 28, 83.
[6] P. Job, *Ann. Chim*., **1936**, 11, 97.
[7] S.E. Turner, R.C. Anderson, *J. Amer. Chem. Soc.*, **1949**, 71**,** 912.
[8] R.C. Dubey, D.K. Maheswari, S. Chand & Company Ltd., **2002**, 172.
[9] B.K. Kumar, V. Ravinder, G.B. Swamy, S.J. Swamy, *Indian. J. Chem*., **1994**, 33, 136.
[10] P.P. Dholakiya, M.N. Patel, *J. Ind. Council. Chem*., **2003**, 20(2), 21.
[11] S. Aryane, N. Sultana, U. Haroon, M.A. Mesaik, *Bioinorg. Chem. Appl*., **2009**.
[12] V. Kumar, R. Dhakrey, *J. Ind. Council. Chem*., **2003**, 20(1), 61.
[13] P. Hongxia, L. Sheming, S. Yuxi, W. Xinato, D. Wenxin, *Cryst. Res. Technol*., **2006**, 41(4), 423.
[14] A. Athar, A.K. Khattak, W. Batool, F. Ullah, Zia ui Haq, M.A. Ullah, J. Khuram, F. Ahmed, *Moroccan. J. Chem.*, **2016**, 4(4), 945.
[15] K. Shankar, R. Roshni, K. Sravankumar, P.M. Reddy, Y. Peng, V. Ravinder, *J. Ind. Chem. Soc*., **2009**, 86, 153.
[16] M.L.H. Nair, L. Sharma, *J. Ind. Chem. Soc*., **2009**, 86, 133.
[17] V. Reddy, N. Patil, B.R. Patil, *J. Ind. Council. Chem*., **2006**, 23(2), 1.
[18] M.K. Zaman, M.S. Arayne, N. Sultana, A. Farooq, *Pak. J. Pharm. Sci*., **2006**, 19(2), 114.
[19] J.R. Dyer, Prentice Hall of India Pvt. Ltd., **1966**.
[20] N. Raman, S. Esthar, J. Thangaraja, *J. Chem. Sci.*, **2004**, 116(4), 209.
[21] D. Prakash, C. Kumar, S. Prakash, A.K. Gupta, K.R.P. Singh, *J. Ind. Chem. Soc*., **2009**, 86, 1257.
[22] P. Singh, D.P. Singh, R. Dwivedi, R. Dhakrey, *J. Ind. Council. Chem*., **2003**, 20(1), 52.
[23] I.P. Kostava, I.L. Manolov, M.K. Rudolova, *Acta. Pharm*., **2004**, 52, 37.
[24] M.M. Hania, *Euro. J. Chem*., **2009**, 6S1, S508.
[25] S. Ghosh, *Der. Pharma. Chemica.*, **2013**, 5(3), 232.