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Der Chemica Sinica, 2014, 5(5):61-66



Complexation of Ni and VO metals with bidentate schiff base derived from Sulfamethoxazole drug

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ABSTRACTS

Schiff bases and their complexes are widely studied owing to their increasing role in biological systems. Schiff bases derived from sulfa drugs act as good ligand to transition metal ions and they exhibit several coordination modes. The metal complexes of Ni(II) and VO(II) have been synthesized with Schiff base of sulfamethoxazole [4-amino-N-(5-methyl-3-isoxazolyl) benzenesulfonamide] and salicylaldehyde. Sulfa drugs concentrate in the urine before being excreted and treat urinary tract infections. Conductometric titrations have suggested metal-ligand ratio of 1:2 for both Ni(II) and VO(II) complexes. The ligand behaves as a bidentate with N, O donor atoms. Complexes have been synthesized and characterized by elemental analysis, IR and UV-Visible spectral studies.

Key words: Sulfamethoxazole, Schiff base, Complex, IR, UV-Visible.

INTRODUCTION

Sulfa drugs are important class of therapeutic compounds and in combination with some other drugs they are used for treatment of various bacterial infections [1]. Metal complexes of Schiff bases have played a pivotal role in the field of coordination chemistry [2]. Study of the coordination behavior of Sulfa drugs is of considerable interest because coordination of metal ion is reported to augment the biological activity of organic compounds. The metal complexes of Schiff bases derived from Sulfa drugs have gained considerable importance due to their pronounced biological activity [3-8]. Schiff base compounds which contain the azomethine (imine) group ($-RC=N-$) are usually prepared by the condensation of a primary amine with an active carbonyl compound [9]. It has been often used as chelating agents (ligands) in the field of coordination chemistry and Schiff base metal complexes were of great interest for many years. It is well known that O and N atoms play a key role at the active sites of numerous metallo biomolecules in the coordination with metals [10].

Here in this paper we are describing synthesis and characterization of Ni(II) and VO(II) complexes with sulfamethoxazole [4-amino-N-(5-methyl-3-isoxazolyl)benzenesulfonamide] Schiff base. (Fig. 1).

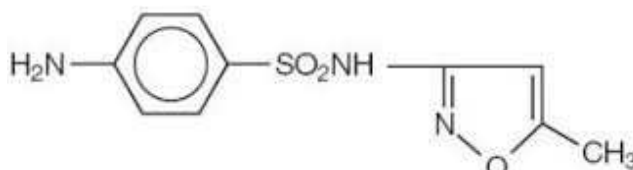


Fig.1. Structure of Sulfamethoxazole

MATERIALS AND METHODS

(a) Reagents and Instruments:

All chemicals and solvents used were of analytical grade. Pure sample of Sulfamethoxazole, molecular formula C₁₀H₁₁N₃O₃S and molecular weight 253.28, was obtained from HiMedia Laboratories Pvt. Ltd. Mumbai. Salicylaldehyde from Central Drug House, New Delhi. Metal salts NiCl₂.6H₂O and VOSO₄.5H₂O were of Merck chemicals.

Elemental analysis (C, H, N and S) were carried out using micro analytical technique on C,H,N,S,O Elemental analyzer at SAIF, CDRI Lucknow. The Infrared spectra of ligand and metal complexes were recorded on KBr pellets in the range 4000-450 cm⁻¹ on Perkin Elmer FTIR spectrophotometer. The NMR spectra were recorded on Bruker Avance II 400 FT NMR spectrometer in DMSO using TMS as the internal standard. Melting points were recorded using melting point apparatus and found 180°C for Schiff base, 198 °C for Ni(II) complex and 232°C for VO(II) complex.

(b) Synthesis of Schiff base:

Equimolar (0.01) solutions of pure drug (0.2532g) and salicylaldehyde (0.12ml) were separately dissolved in methanol water mixture (1:1) and refluxed for four hours and kept for a day. Pale yellow crystals of sulfamethoxazole Schiff base (SMZ-SD) were formed in the reaction mixture which were filtered and washed thoroughly with 50% methanol–water mixture, dried over vacuum and weighed. Melting point of Schiff base was recorded. (Fig. 2).

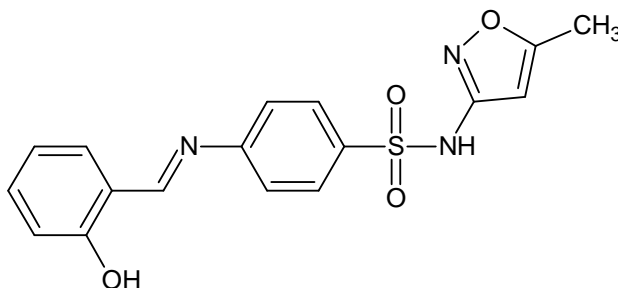


Fig. 2. Structure of Sulfamethoxazole-Salicylalimine (Schiff Base)

(c) Ligand-Metal ratio:

For the synthesis of complexes, we need to confirm metal-ligand ratio by conductometric titration method. 0.01 M solution of Sulfamethoxazole-Salicylalimine Schiff base was prepared in 60:40 mixture of acetone and distilled water. Similarly, solution of metal salts NiCl₂.6H₂O and VOSO₄.5H₂O were prepared in the same solvent of 0.02 M concentration. 20 ml of ligand solution was diluted to 200 ml. The ligand was titrated against metal salt solutions and conductance was recorded after each addition of metal salt solution. From the graph between corrected conductance and volume of titrant added, it was concluded that the complex formation has taken place in the ratio of 1:2 (metal-ligand).

(d) Synthesis of complex:

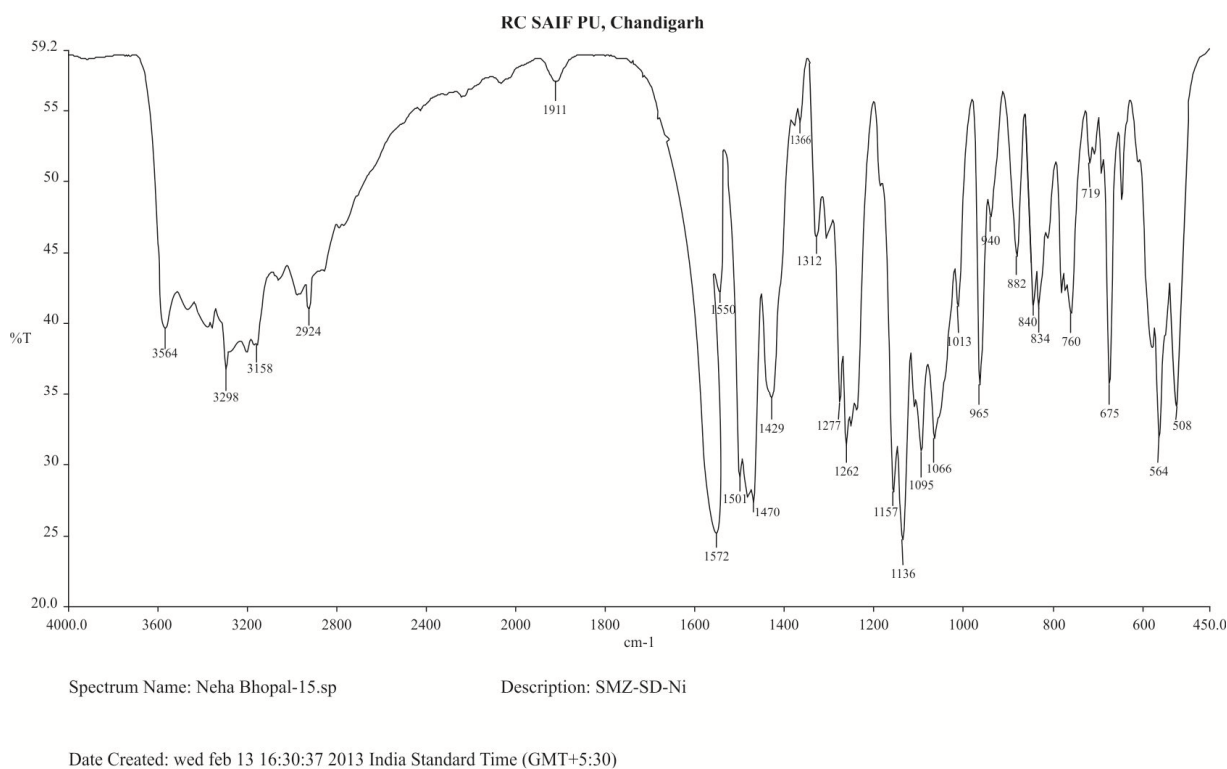
For the synthesis of complexes, 0.02 mol sulfamethoxazole–salicylalimine (L) solution was prepared in 60% acetone–water solvent and refluxed for four hours with 0.01 mol solution of metal salts separately. The refluxed solutions were kept for some days. Solid crystalline compounds appeared in the solution, which were filtered, washed with 60% acetone–water mixture, dried and weighed. Melting point of the complexes was recorded.

RESULTS AND DISCUSSION

The analytical data of the complexes are given in Table 1. All these complexes are analyzed for 1:2 stoichiometry of the type ML₂. On the basis of these characterizations it has been found that all the complexes are non-hygroscopic, stable at room temperature, insoluble in water but fairly soluble in DMSO.

(a) Infrared Spectral studies:

The relevant spectral bands and their assignments are recorded. The IR spectra [11-12] of the complexes indicate that the ligand behaves as bidentate and the metal coordinates via azomethine nitrogen and phenolic –OH groups. The shift of $\nu_{C=N}$ to lower wave number by 20-30 cm^{-1} in the complexes indicates that these groups are involved in complexation [13]. The ligand shows strong band at 3430 cm^{-1} due to phenolic –OH group [14]. This band is absent in all the metal complexes indicating the involvement of this group in complex formation [15]. Moreover, the shift of the ν_{C-O} phenolic bands from 1282 cm^{-1} in ligand to 1312–1313 cm^{-1} in the spectra of metal complexes supports the coordination of the phenolic oxygen atom to the metal ion [16]. The Ni(II) complex shows a broad band in the region 3564 cm^{-1} suggesting the presence of coordinated water [17,18]. The bands for ν_{M-O} mode [19] appeared in the range of 546–564 cm^{-1} in both the complexes. The presence of sharp band in the region 507–508 cm^{-1} in the spectra of the complexes assigned to ν_{M-N} mode [20], further support the involvement of nitrogen atom in coordination. The VO(II) complex shows a small band in the region 954 cm^{-1} suggesting the presence of M=O bond. (Fig. 3 & 4).

**Fig. 3. IR Spectra of SMZ-SD-Ni**

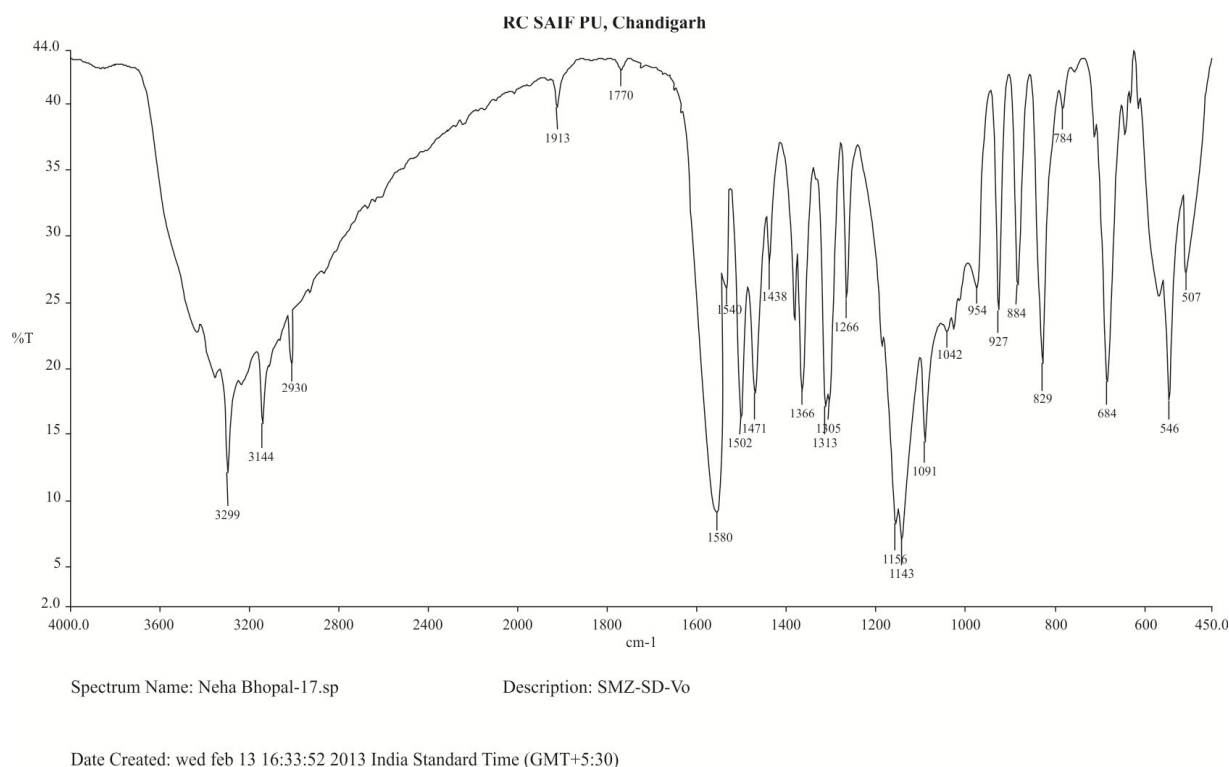


Fig. 4. IR Spectra of SMZ-SD-VO

(b) UV-Visible Spectral studies:

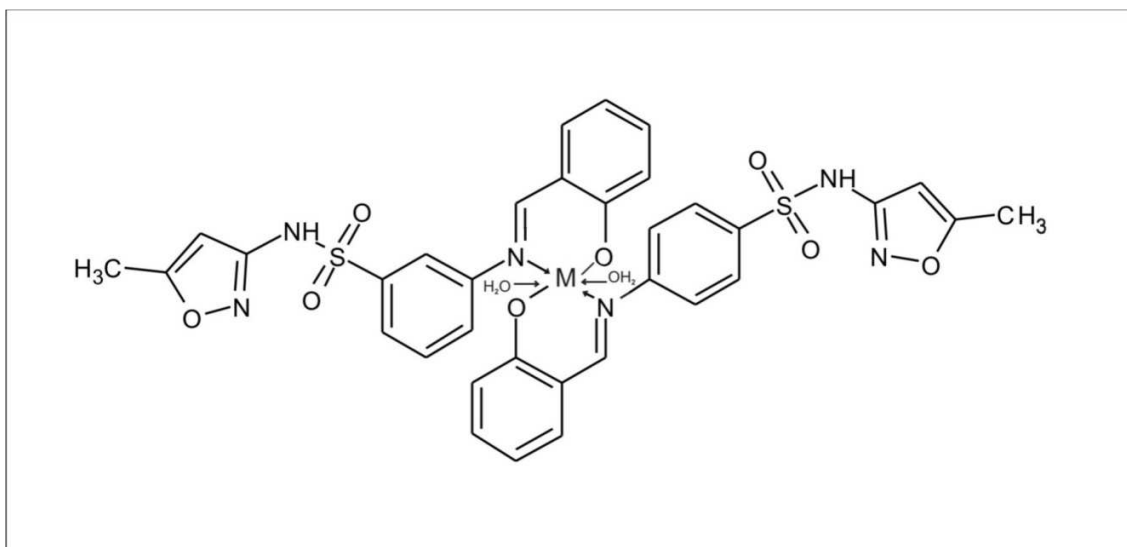
The Ni(II) complex shows magnetic moment value 2.80 B.M. The electronic spectra of the Ni(II) complex shows bands at 10259cm^{-1} , 17197cm^{-1} and 23585cm^{-1} which are corresponding to spin allowed transitions ${}^3T_{2g}(F) \leftarrow {}^3A_{2g}(F)$, ${}^3T_{1g}(F) \leftarrow {}^3A_{2g}(F)$ and ${}^3T_{1g}(P) \leftarrow {}^3A_{2g}(F)$ suggesting octahedral geometry of the complex around the metal ion. The electronic spectra of VO(II) complex shows bands at 13986cm^{-1} , 17123cm^{-1} and 21786cm^{-1} assigned ${}^2E \leftarrow {}^2B_2$, ${}^2B_1 \leftarrow {}^2B_2$ and ${}^2A_1 \leftarrow {}^2B_2$ transitions respectively indicating square pyramidal geometry. The value of μ_{eff} for oxovanadium complex is 1.75 B.M., also support the square pyramidal geometry.

Table-1. Analytical data of the ligand and complex

S. No.	Composition of ligand / complexes (m.wt.)	Color	Yield (%)	m.p. ($^{\circ}\text{C}$)	Elemental analyses(%):found (calcd)				
					C	H	N	S	M
1.	C17H15N3O4S	Pale yellow	68	180	56.43 (57.13)	4.13 (4.23)	11.50 (11.76)	8.91 (8.97)	-
2.	C34H32N6O10S2Ni	Pista Green	65	198	50.29 (50.57)	3.66 (3.99)	10.21 (10.41)	8.01 (7.94)	7.21 (7.27)
3.	C34H32N6O10S2VO	Yellow	61	232	51.48 (52.38)	3.68 (3.62)	10.87 (10.78)	8.37 (8.22)	6.69 (6.53)

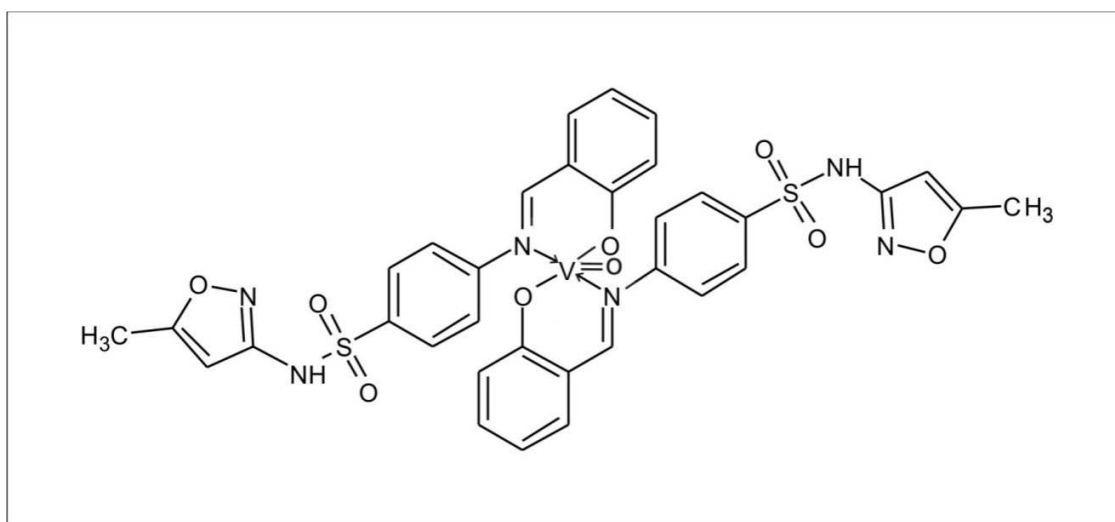
CONCLUSION

From the analytical and spectral data, it can be concluded that the synthesized complexes are stable with probably following structures. (Fig. 5 & 6).



[M=Ni]

Fig. 5. Structure of SMZ-SD-Ni complex



[M=VO]

Fig. 6. Structure of SMZ-SD-VO complex

Acknowledgments

The authors owe their sincere thanks to UGC for sanctioning UGC Research Award to Dr. Suman Malik, one of the coauthors. Authors are also indebted to CDRI Lucknow for providing the facilities of elemental analysis, SAIF Chandigarh for IR and UV-Visible analysis

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