

## Research Article

# Synthesis, Characterization, and Biological Evaluation of Some 3d-Metal Complexes of Schiff Base Derived from Xipamide Drug

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Received 31 August 2012; Revised 25 October 2012; Accepted 7 November 2012

Academic Editor: Alfonso Castiñeiras

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The present paper deals with the synthesis and characterization of metal complexes of Schiff base derived from xipamide, a diuretic drug. The bidentate ligand is derived from the inserted condensation of 5-aminosulfonyl-4-chloro-N-2,6-dimethyl phenyl-2-hydroxybenzamide (Xipamide) with salicylaldehyde in a 1 : 1 molar ratio. Using this bidentate ligand, complexes of Hg(II), Zn(II), and VO(IV) with general formula  $ML_2$  have been synthesized. The synthesized complexes were characterized by several techniques using molar conductance, elemental analysis, magnetic susceptibility, FT-IR spectroscopy, electronic spectra, mass spectra, and particle size analysis. The elemental analysis data suggest the stoichiometry to be 1 : 2 [M : L]. All the complexes are nonelectrolytic in nature as suggested by molar conductance measurements. Infrared spectral data indicate the coordination between the ligand and the central metal ion through deprotonated phenolic oxygen and azomethine nitrogen atoms. Spectral studies suggest tetrahedral geometry for Hg(II), Zn(II) complexes, and square pyramidal geometry for VO(IV) complex. The pure drug, synthesized ligand, and metal complexes were screened for their antifungal activities against *Aspergillus niger* and *Aspergillus flavus*. The ligand and its Hg(II) and VO(IV) complexes were screened for their diuretic activity too.

## 1. Introduction

Coordination complexes are gaining importance in recent years especially in the designing of long acting drugs in metabolism. The metal complexes from bidentate ligands have often been studied recently because of their technical applications [1, 2] and applications in enhancement of drug action [3, 4]. Transition metals are essential for normal functioning of living organism and are, therefore, of great interest as potential drugs [5]. The coordination chemistry of nitrogen donor ligands is an active area of research. A great deal of attention in this area has been focused on the complexes formed by 3d metals with bidentate ligands using both sulfur and nitrogen [6, 7]. The Schiff bases are an important class of ligands in coordination chemistry. The study of structural and binding features of various Schiff base complexes can play an important role in better understanding of the complex biological process. Schiff bases derived from salicylaldehyde are well known for their interesting ligational properties and exclusive applications in different fields

[8–10]. It is well known from the literature that Schiff bases derived from thiazide drugs have a strong ability to form metal complexes [11]. The interaction of these donor ligands and metal ions gives complexes of different geometries, and literature survey reveals that these complexes are potentially more biologically active. Thus, in recent years Schiff bases and their metal complexes have attained much attraction because of their extensive biological activities [12, 13]. Keeping the above fact in our mind and in continuation of our earlier work on transition metal complexes with Schiff bases [14, 15], the ligand xipamide-salicylaldehyde Schiff base (L) has been synthesized. In the present paper, the synthesis and characterization of the ligand and its complexes with Hg(II), Zn(II), and VO(IV) are being reported.

## 2. Experimental

All the chemicals used were of AR/GR grade and purchased from E-Merck (USA). Chemicals were used without any

TABLE 1: Analytical data and molar conductance values for ligand and metal complexes.

S. number	Ligand/complexes	Elemental analysis (%): found (Calcd.)					M	M.pt. (°C)	Color	Molar conductance $\Omega^{-1} \text{ cm}^2 \text{ mol}^{-1}$
		C	N	S	Cl	M				
1	L	56.87 (57.57)	5.84 (5.96)	6.91 (6.97)	7.50 (7.61)	—	250	Peach	—	
2	HgL <sub>2</sub>	47.08 (47.32)	4.97 (5.01)	5.48 (5.73)	6.34 (6.36)	17.70 (17.98)	241	Off-White	13.2	
3	ZnL <sub>2</sub>	53.48 (53.85)	5.64 (5.71)	6.38 (6.52)	7.01 (7.14)	6.51 (6.67)	218	White	12.8	
4	VO <sub>2</sub>	52.87 (53.17)	5.79 (5.70)	6.67 (6.52)	7.09 (7.25)	5.27 (5.18)	242	Yellow	11.8	

purification. Xipamide drug was provided by Dishman Pharmaceuticals which was used as such for the synthesis of ligand.

Elemental analyses were carried out on model 240 PerkinElmer elemental analyzer at CDRI, Lucknow. Metal contents were determined gravimetrically using standard methods [16]. Conductivity measurements were made in anhydrous DMF on a Systronics model 305 (India) Conductivity Bridge. Magnetic susceptibility measurements of the complexes in the solid state were determined by vibrating sample magnetometer at Centre for Advance Technology, Indore at room temperature. The electronic spectra of the metal complexes in DMF were recorded on a Perkin-Elmer UV WinLab Spectrophotometer at School of Studies in Chemistry and Biochemistry, Vikram University, Ujjain, India. The infrared spectra of the ligand and complexes were recorded in KBr pellets using Perkin-Elmer FT-IR spectrophotometer in the range 4000–400  $\text{cm}^{-1}$  at School of Studies in Chemistry and Biochemistry, Vikram University, Ujjain, India. Particle size analysis was carried out at SICART, Gujarat using, laser diffraction particle size analyzer. The instrument used was CILAS 1064L/D model in the range of 0.04–500  $\mu\text{m}$ . The melting points of the ligand and complexes were recorded in open capillaries on a capillary melting point apparatus.

The antifungal activities of both the ligands and their complexes were tested *in vitro* for growth inhibitory against *Aspergillus niger* and *Aspergillus flavus* by agar growth food poison technique [17] at different concentrations compared with Grisofulvin as appositive control.

The diuretic activity of Hg(II) and VO(IV) complexes were tested on white albino mice at Jawaharlal Nehru Cancer Hospital and Research Centre, Bhopal, India. Mice were fed with standard rodent pellet diet and acidified double-distilled water. The selected test compound, ligand, and standard drug xipamide (XM) at a dose of 0.24 mg suspended in 0.5% gum acacia were administrated to animals of respective group. One group of animal was taken as control. For the measurement of weight of urine, Whatman no. 1 filter paper was used. The urine weight was recorded after two hours.

**2.1. Synthesis of the Ligand (Xipamide Salicylaldehyde).** Equimolar (0.01 M) solutions of pure drug (0.22 gm) and salicylaldehyde (0.14 mL) were separately dissolved in methanol-water mixture (1:1) and refluxed for four hours and kept for a day. Peach colour crystals of xipamide Schiff base were formed in the reaction mixture and were filtered and washed thoroughly with 50% methanol-water mixture, dried

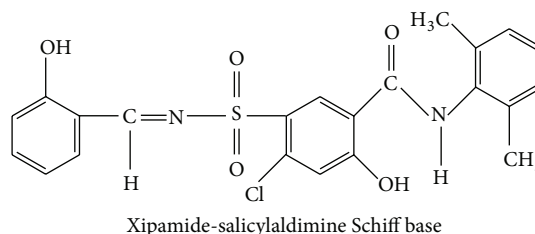


FIGURE 1: Structure of ligand.

over vacuum, and weighed. Melting point of Schiff base was recorded. The structure of the synthesized ligand is shown in Figure 1.

**2.2. Synthesis of Complexes.** For the synthesis of complexes, 0.006 M ligand solution was prepared in 50% acetone-water solvent and refluxed for four hours with 0.003 M 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 50% acetone-water mixture, dried, and weighed. Melting points of the complexes were recorded.

### 3. Results and Discussion

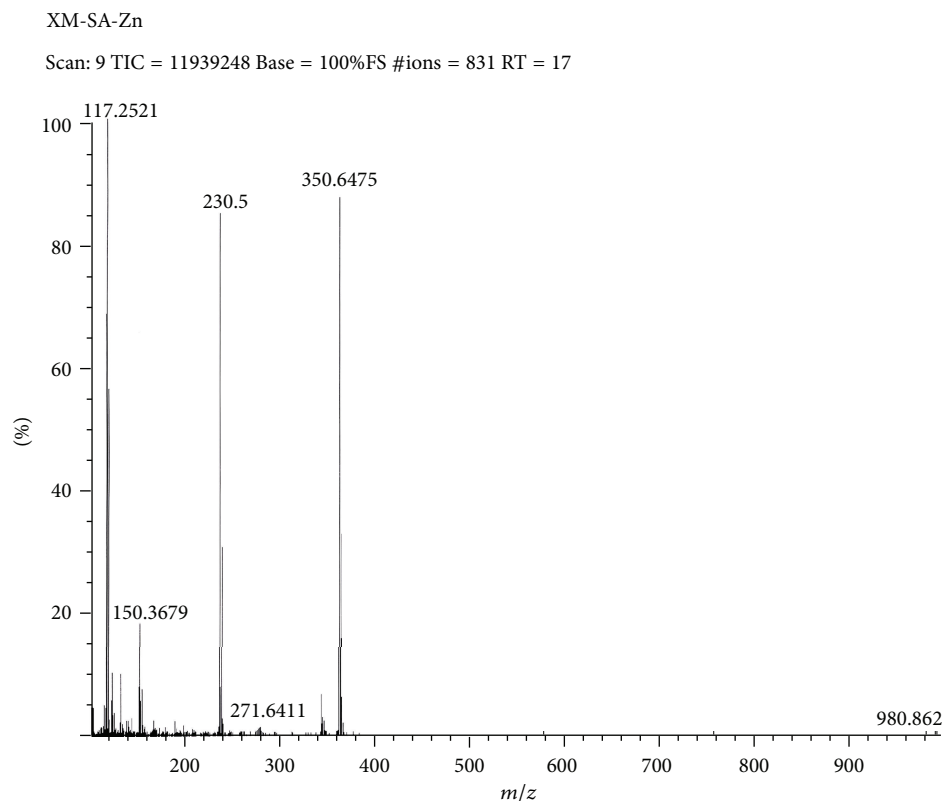
The analytical data of the complexes and their molar conductance values are given in Table 1. All these complexes are analyzed for 1:2 stoichiometry of the type  $\text{ML}_2$ . On the basis of these characterizations it has been found that all the complexes are nonhygroscopic, stable at room temperature, insoluble in water, but fairly soluble in DMSO. The molar conductance values of these complexes are too low to account for their electrolytic behavior [18, 19].

#### 3.1. Spectral Studies of Ligand and Its Complexes

**3.1.1. IR Spectra.** The characteristic vibrations and assignments of ligand and its complexes are described in Table 2. The IR spectra of the complexes indicate that the ligand behaves as bidentate and coordinates with metals via azomethine nitrogen and phenolic  $-\text{OH}$  groups. The shift of  $\nu_{\text{C}=\text{N}}$  to lower wave number by 30–40  $\text{cm}^{-1}$  in the complexes indicates that these groups are involved in complexation [20, 21]. The ligand shows strong band at 3386  $\text{cm}^{-1}$  due to phenolic  $-\text{OH}$  group [22]. This band is absent in all the metal complexes indicating the involvement of this group

TABLE 2: IR spectral data ( $\text{cm}^{-1}$ ) of ligand and its complexes.

S. number	Ligand/complexes	$\nu_{\text{N-H}}$	$\nu_{\text{C=N}}$	$\nu_{\text{C-O}}$	$\nu_{\text{C=O}}$	$\nu_{\text{M-N}}$	$\nu_{\text{M-O}}$
1	$\text{C}_{22}\text{H}_{19}\text{N}_2\text{O}_5\text{ClS}$	3302	1639	1282	1671	—	—
2	$\text{C}_{44}\text{H}_{36}\text{N}_4\text{O}_{10}\text{Cl}_2\text{S}_2\text{Hg}$	3301	1598	1312	1669	514	615
3	$\text{C}_{44}\text{H}_{36}\text{N}_4\text{O}_{10}\text{Cl}_2\text{S}_2\text{Zn}$	3300	1623	1288	1681	514	580
4	$\text{C}_{44}\text{H}_{36}\text{N}_4\text{O}_{10}\text{Cl}_2\text{S}_2\text{VO}$	3300	1620	1281	1680	505	608

FIGURE 2: Mass spectrum of  $\text{ZnL}_2$ .

in complex formation [23]. Moreover, the shift of the  $\nu_{\text{C-O}}$  phenolic bands from  $1282\text{ cm}^{-1}$  in ligand to  $1282\text{--}1312\text{ cm}^{-1}$  in the spectra of metal complexes supports the coordination of the phenolic oxygen atom to the metal ion [24]. The bands for  $\nu_{\text{M-O}}$  modes appeared in the range of  $580\text{ cm}^{-1}\text{--}615\text{ cm}^{-1}$  in all the complexes [25]. The presence of sharp band in the region  $503\text{--}514\text{ cm}^{-1}$  in the spectra of all the complexes assigned to  $\nu_{\text{M-N}}$  mode [26] further support the involvement of azomethine nitrogen atom in coordination. In addition to these bands VO(IV) complex exhibits the characteristic stretching frequency [27, 28] for  $\text{V=O}$  at  $952\text{ cm}^{-1}$ .

**3.1.2. Magnetic Susceptibility and Electronic Spectra.** The magnetic moment obtained for the oxovanadium (IV) complex at room temperature is 1.77 B.M, and its electronic spectra displayed bands at 13986, 17094, and  $21740\text{ cm}^{-1}$  assigned for  ${}^2\text{B}_2 \rightarrow {}^2\text{E}$ ,  ${}^2\text{B}_2 \rightarrow {}^2\text{B}_1$ , and  ${}^2\text{B}_2 \rightarrow {}^2\text{A}_1$  transitions, respectively. These values suggest the square pyramidal geometry for VO(IV) complex [29]. The Hg(II) and Zn(II)

complexes are found to be diamagnetic as expected for  $d^{10}$  systems and may have tetrahedral geometry [30].

**3.1.3. Mass Spectra.** The mass spectrum of  $[\text{Zn}(\text{C}_{22}\text{H}_{18}\text{ClN}_2\text{O}_5\text{S})_2]$  as shown in Figure 2 shows a molecular ion peak at  $m/z$  980 (I) due to  $[\text{Zn}(\text{L})_2]^+$  which is in accordance with the proposed formula of the complex. The other peaks at  $m/z$  values 151 and 271 may be due to the fragments  $(\text{C}_9\text{H}_{11}\text{NO})^+$  and  $[\text{Zn}(\text{ClC}_6\text{H}_4\text{COH})\text{SO}_2\text{NH}]^+$ . The base peak at  $m/z$  117 may be due to the zinc metal linked to the donor atoms of the ligand. Such type of fragmentation pattern has been reported by many workers [31, 32].

**3.1.4. Particle Size Analysis.** To find out the maximum efficiency of the drugs and their metal complexes, studies on the particle size analysis are being considered very helpful [33]. The bioavailability of low-solubility drug is often intrinsically related to the drug particle size. Smaller particle size of the complexes is responsible for the enhanced solubility of the drug [34]. The results of the particle size analysis carried

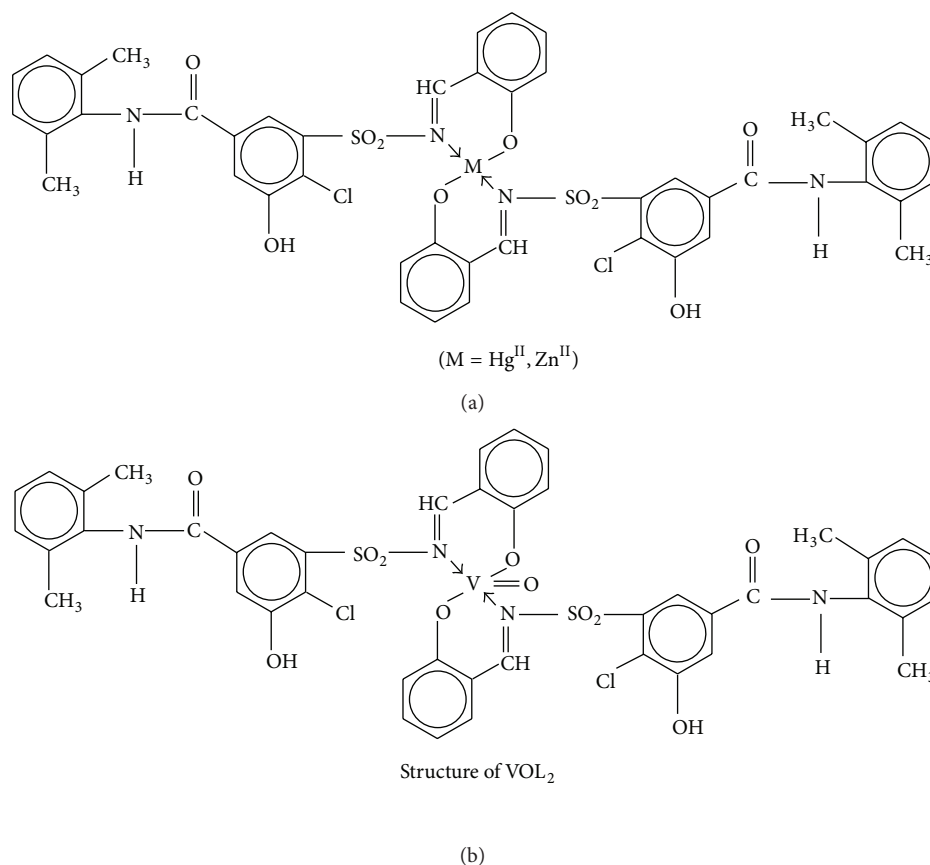


FIGURE 3: Structure of metal complexes.

TABLE 3: Particle size measurement of pure drug, ligand and its metal complexes.

S. number	Sample code	Particle size ( $\mu\text{m}$ )
1	XM	112
2	L	95.6
3	HgL <sub>2</sub>	67.5
4	VOL <sub>2</sub>	64.5

out for the pure drug, ligand, and its Hg(II) and VO(IV) complexes have been recorded in Table 3. These results reveal that, after complexation, the size of the ligand and complexes got reduced to a considerable extent as compared to their parent drug xipamide (XM). It is well known that smaller size leads to better absorption, so it is concluded from our result that complexation enhanced the absorption and potency of the drug [35].

**3.1.5. Antimicrobial Activity.** For antifungal activity the ligand and its metal complexes were screened against *Aspergillus niger*, and the findings are given in Table 4. These complexes showed higher activity with 12–17 mm inhibition than the ligand which showed only 11.48 mm inhibition at the same concentration as that of the test drug. However, ligand

TABLE 4: Antimicrobial screening data of the ligand L and its metal complexes.

S. number	Compound/ complex	Antifungal zone of inhibition (in mm)	
		<i>A. niger</i>	<i>A. flavous</i>
1	L	11.48	10.38
2	HgL <sub>2</sub>	15.76	16.41
3	ZnL <sub>2</sub>	13.45	14.33
4	VOL <sub>2</sub>	17.00	21.22
5	Grisofulvin	19.38	18.22

and their complexes showed lower activity as compared to standard drug griseofulvin with 19.38 mm inhibition at the same concentration.

Antifungal activity studies of ligand and its complexes against *Aspergillus flavus* showed that all the complexes with 11.47–21.22 mm inhibition showed higher activity than the ligand which showed only 10.38 mm inhibition only. VO(IV) complex showed higher activity than standard drug which showed 18.22 mm inhibition at the same concentration as that of the test drug. It is known that chelation tends to make the ligand act as more powerful and potent antimicrobial agent, thus inhibiting more of the microbes than the parent ligand [36].

TABLE 5: Diuretic screening data of the pure drug, ligand, and its metal complexes.

Group	Ligand/complexes	Average volume of urine ( $\mu\text{L}$ )			Final average ( $\mu\text{L}$ )
		1:00 PM	3:00 PM	5:00 PM	
I	Control	113.94	167.22	118.01	133.06 $\pm$ 29.656
II	Xipamide	155.66	208.09	266.91	210.22 $\pm$ 55.656
III	L	175.75	235.48	289.10	233.44 $\pm$ 56.702
IV	HgL <sub>2</sub>	265.54	283.12	343.12	297.26 $\pm$ 40.6777
V	VOL <sub>2</sub>	386.66	375.29	378.57	380.17 $\pm$ 5.852

**3.1.6. Diuretic Activity.** The diuretic activity of the Hg(II) and VO(IV) complexes was tested on white albino mice and recorded in Table 5. The selected test compound, ligand, and standard drug xipamide at a dose of 0.24 mg suspended in 0.5% gum acacia were administered to animals of the respective group. The result revealed that metal complexes have more diuretic activity as compared to the respective ligand and pure drug. Thus it is revealed that complexation can increase the pharmaceutical activity of the parent drug [37].

Statistical analyses were performed by using one-way analysis of variance ANOVA followed by GraphPad InStat 3. Statistical significance was accepted at the 5% level ( $P < 0.05$ ). The VO(IV) complex is found to be more significant with  $P < 0.001$  whereas Hg(II) complex is also found to be significant with  $P < 0.05$ .

#### 4. Conclusion

The outcome of the above results confirms the stoichiometry of the complexes to be 1:2 [M:L] as indicated by elemental analysis and conductometric measurements. IR spectra suggest that the ligand behaves as bidentate and coordinates to the central metal ion through azomethine nitrogen and phenolic -OH group. This has been further confirmed on the basis of NMR spectral studies. Mass spectra further support the above stoichiometry on the basis of respective molecular masses and fragmentation patterns. Thus, on the basis of above physicochemical and spectral studies, the assigned structures for the metal complexes are shown in Figures 3(a) and 3(b). The complexes are found to have higher biological activities as compared to the respective ligand and the parent drug that, somehow, justifies the purpose of the research work. The present work will be further extended to the synthesis of metal complexes using other biologically active metals and evaluation of their biological activities.

#### Acknowledgments

The authors gratefully acknowledge the financial support provided by UGC. They are also indebted to CDRI, Lucknow, for providing the facilities of elemental analysis, SICART Gujrat for TGA and particle size studies, IIT Chennai for mass spectra, and Vikram University Ujjain for recording IR and electronic spectra. The authors are thankful to Dr. N. Ganesh, Senior Research in Charge and Scientist of Jawaharlal Nehru Cancer Hospital and Research Centre, Bhopal, India, for developing protocol and facilities for diuretic activity.

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