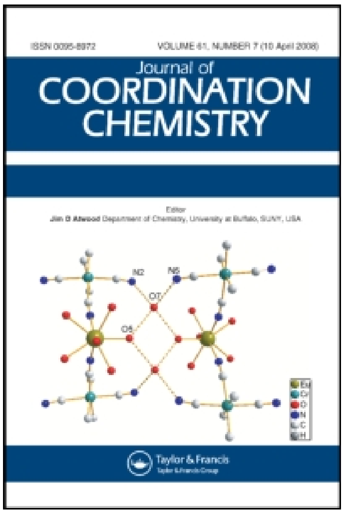

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Synthesis, spectral characterization, and antimicrobial studies of metal complexes of the Schiff base derived from [4-amino-N-guanylbenzene sulfonamide] and salicylaldehyde

C. D. Sheela[a]; C. Anitha[a]; P. Tharmaraj[b]; D. Kodimunthri[b]
[a] Department of Chemistry, The American College, Madurai-625002, Tamil Nadu, India [b] Department of Chemistry, Thiagarajar College, Madurai-625009, Tamil Nadu, India

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Synthesis, spectral characterization, and antimicrobial studies of metal complexes of the Schiff base derived from [4-amino-N-guanylbenzene sulfonamide] and salicylaldehyde

C.D. SHEELA*†, C. ANITHA†, P. THARMARAJ‡
and D. KODIMUNTHRI‡

†Department of Chemistry, The American College, Madurai – 625002, Tamil Nadu, India
‡Department of Chemistry, Thiagarajar College, Madurai – 625009, Tamil Nadu, India

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Sulfaguanidinesalicylaldimine is a good bacteriostatic and a good complexing agent. Schiff-base complexes of Cu(II), Ni(II), Co(II), Zn(II), Cd(II), VO(IV), and UO_2(VI) have been synthesized. The structural features of the complexes have been confirmed by microanalytical data, FAB mass, IR, UV-Vis, ^{1}H-NMR, and EPR spectra. Molar conductance indicates the presence of nonelectrolytes. Spectroscopic and other analytical studies reveal the square-planar geometry for copper, square-pyramidal geometry for oxovanadium, seven-coordinate UO_2(VI) complex, and octahedral geometry for other complexes. The EPR spectrum of the copper complex in the powdered form at 77 K was recorded. The redox behavior of the copper and oxovanadium complexes was studied using cyclic voltammetry. Antimicrobial activities of the compounds have been studied against microorganisms such as *Escherichia coli, Staphylococcus aureus*, and *Candida* by well-diffusion technique in DMSO. Some of the complexes have higher activity than the free ligand and the standard.

Keywords: Salicylaldehyde; Schiff-base complexes; Antimicrobial activity; Sulfonamide

1. Introduction

Schiff bases are important ligands in coordination chemistry a century since their discovery [1, 2]. Stereochemical studies on Schiff-base complexes of β-diketones and salicylaldehyde have shown that the structures lie between square planar and tetrahedral with other intermediate structures too. Varieties of structures have been interpreted in terms of steric and electronic factors [2]. Comparative studies of salicylaldimines and 2-hydroxychalconimines show that the latter is a weak field ligand [3] since the conjugative interaction reduces the basicity of imine nitrogen. In the case of naphthylchalconimines, conjugation has no effect on stereochemistry due to

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*Corresponding author. Email: cdsheela@yahoo.com

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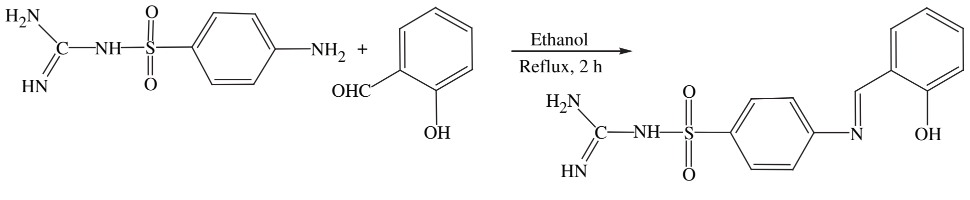
steric interactions [4]. Sulfonamides are used as proton-conducting materials for application in solid state electrochemical devices like smart windows and sensors [5].

Sulfonamides involve a family of products widely known because of their pharmaceutical applications. Apart from being innocuous to human beings, they are efficient against pathogenic germs. Several spectrophotometric methods for determining sulfonamides in pharmaceutical preparation exist [6–8]. Their Schiff bases and complexes were investigated for physiologically relevant carbonic anhydrase [9]. Sulfaguanidine contains the basic sulfonamide skeleton which is active against microorganisms; metalation would either enhance or reduce antimicrobial activity. Interesting structural features and biological activity of such compounds were key factors to carry out this investigation.

Metal complexes of Schiff bases derived from sulfaguanidine have been investigated [10]. Schiff-base metal complexes involving sulfaguanidine and salicylaldehyde are expected to possess enhanced biological activities [1–11]. In this connection, sulfaguanidine condensed with salicylaldehyde offers an extension to the study since perturbation in the ligand backbone may affect the stereochemistry of the complexes. In this work, we synthesized Schiff-base complexes of Cu(II), Ni(II), Co(II), Zn(II), Cd(II), VO(IV), and UO_2(VI), characterized them, and studied their biological activities against various microorganisms. Scheme 1 illustrates the synthesis of the Schiff base.

2. Experimental

All chemicals were of analytical reagent (AR) grade and purified by standard procedures. Molar conductances were measured in DMSO at room temperature (RT) using Systronic Conductivity Bridge 305. IR spectra of the ligand and complexes were recorded using KBr pellets in a Shimadzu spectrophotometer. Electronic spectra of the complexes and ligand in DMSO (10^{-3} M) were recorded in a Jasco-V-530 UV-Vis spectrophotometer. ^{1}H-NMR spectra were recorded in DMSO-d_6 using TMS as the internal standard in a 300 MHz NMR spectrometer at CDRI, Lucknow. FAB mass spectra were recorded on a JEOL SX 102/Da-600 mass spectrometer at RT using NBA matrix with data system using Ar/Xe as FAB gas. Mueller–Hinton agar was used for testing the susceptibility of microorganism to antibacterial agents using the well-diffusion technique with amikacin and ketoconazole as standards. ESR spectra of the copper complex in powdered form at RT and at 77 K were recorded at X-band frequency on a JEOL TES 100 ESR spectrometer using DPPH ($g = 2.0036$)



Scheme 1. Synthesis of the Schiff base (LH).

as an internal field marker. Cyclic voltammetric measurements for Cu(II) and VO(IV) complexes in DMSO were carried out on a CH (CHI 608C Spl) basic electrochemistry system. A three-electrode system consisting of platinum disk working, platinum wire counter, and Ag/AgCl reference electrodes were used for the oxovanadium complex; for the copper complex, the working electrode was a glassy carbon electrode. Tetrabutylammonium tetrafluoroborate was used as the supporting electrolyte. A glass cell with a Teflon cell cover holding counter, working and reference electrode, and deoxygenating purge tube formed the sample cell.

2.1. *Synthesis of the ligand (HL)*

The Schiff base was prepared by mixing a hot solution of sulfaguanidine (20 mM) with a solution of salicylaldehyde (20 mM) in 30 mL of acetone. The mixture was refluxed for 2 h. The resulting solution was concentrated on a water bath and cooled. The solid was separated by filtration, purified by crystallization from acetone, and dried in vacuum over anhydrous calcium chloride. The ligand was obtained as yellow crystals in 80% yield.

2.2. *Synthesis of metal complexes*

Metal complexes of the Schiff base were prepared by the addition of 15 mL warm aqueous solution of metal chloride or sulfate or nitrate (10 mM) to the warm solution of the ligand (20 mM) dissolved in 10 mL acetone and adjusting pH to 8 by adding 1 M NaOH. The mixture was digested for a few minutes on the water bath and the solid complexes were collected by filtration, washed with a small volume of methanol and ether, and dried under vacuum.

2.3. *Synthesis of VO(IV) and UO2(VI) complexes*

Warm 15 mL aqueous solution of $VOSO_4$ or UO_2Cl_2 (10 mM) was added to a warm solution of the ligand (20 mM) dissolved in 10 mL acetone and the pH was adjusted to 8 by adding 1 M NaOH. The mixture was stirred under reflux for 12 h, the precipitated complexes were collected by filtration, washed with 1 : 1 ethanol : water, and dried in vacuum. The above detailed preparation is given as an example and other complexes were obtained similarly.

3. Results and discussion

The metal complexes are stable at RT and insoluble in water but soluble in DMF, DMSO, and acetone. The molar conductances of these complexes in 10^{-3} M DMSO are in the range of 14.1–15.8 ($\mathrm{S\,cm^{-2}\,M^{-1}}$), consistent with nonelectrolytes [12].

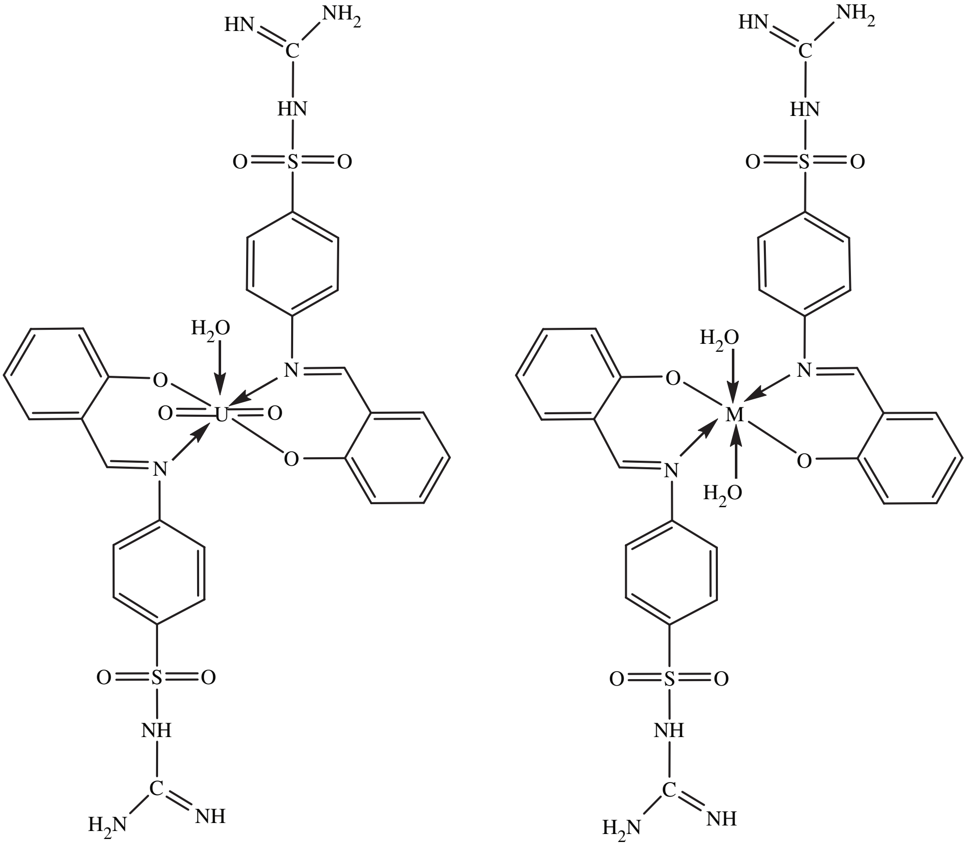


Figure 1. Proposed structure of the metal complexes M = Ni(II), Co(II), Zn(II), Cd(II), and Mn(II).

The analytical data of the ligand and complexes, collected in table 1, agree with the proposed formula ML_2 for Cu(II), VO(IV), and $ML_2 \cdot H_2O$, for UO_2(VI) and $ML_2 \cdot 2H_2O$, and for Ni(II), Co(II), Zn(II), and Cd(II) (figure 1).

3.1. *IR spectra*

IR stretching frequencies of the ligands and its metal complexes are shown in "Supplementary material". The azomethine stretching vibration in free ligand occurs at $1575\,cm^{-1}$. Shifting of the band to lower wavenumbers ($1529–1533\,cm^{-1}$) in the complexes indicates the participation of the azomethine in coordination [12–14]. The phenolic ν(C–O) is at $1431\,cm^{-1}$ in the free ligand, but appears at higher frequencies in the complexes ($1432–1499\,cm^{-1}$), confirming the involvement of phenolic oxygen in complex formation [15].

The ligand exhibits two bands in the NH stretching region $3230–3300\,cm^{-1}$ and an NH deformation band around $1620\,cm^{-1}$. The ν(NH) and $\nu(NH_2)$ of sulfonamide/amino in the uncoordinated Schiff base remains mostly unchanged in spectra of the complexes, suggesting that the sulfonamide nitrogen/amino group do not take part

Table 1. Elemental analyses of the ligand and metal complexes.

	Analysis found (Calcd) (%)								
Compound	Formula weight	M	C	H	N	S	Yield (%)	Melting point (°C)	Λ_M ($S\,cm^2\,M^{-1}$)
LH	319.35	–	52.64 (52.83)	4.23 (4.40)	17.78 (17.61)	9.75 (10.06)	91	124	–
CuL_2	698.23	9.06 (9.11)	47.97 (48.16)	3.72 (4.01)	16.08 (16.06)	8.88 (9.18)	85	293	13.31
$CoL_2(H_2O)_2$	674.75	8.21 (8.08)	45.82 (46.09)	4.07 (4.39)	15.64 (15.36)	8.91 (8.21)	82	245	14.08
	733.68								
$NiL_2(H_2O)_2$	733.44	8.12 (8.06)	46.04 (46.11)	4.13 (4.39)	15.25 (15.37)	8.37 (8.78)	83	278	11.20
$ZnL_2(H_2O)_2$	740.16	8.15 (8.84)	45.67 (45.71)	4.16 (4.35)	14.92 (15.23)	8.92 (8.71)	87	293	10.40
$CdL_2(H_2O)_2$	787.16	13.94 (14.32)	42.85 (43.00)	3.93 (4.09)	14.21 (14.32)	8.02 (8.18)	80	240	12.20
$UO_2L_2(H_2O)$	922.36	25.75 (26.80)	36.33 (37.46)	3.41 (3.52)	12.02 (12.15)	6.89 (6.95)	84	253	11.30
VOL_2	701.60	7.13 (7.26)	47.84 (47.93)	3.67 (3.73)	15.89 (15.97)	9.06 (9.14)	80	260	09.10

in coordination. Bands at 1130 and 1300 cm^{-1} are attributed to symmetric and asymmetric stretching vibrations, respectively, of the sulfone group, ν(O=S=O) in the free ligand [12, 16, 17]; these bands remain unchanged in the complexes suggesting that sulfonamide oxygen is not involved in coordination. Previous studies on complexes of sulfa-drugs [10] indicated that the SO_2 modes of the ligands appear as sharp bands at 1306–1364 and 1138–1178 cm^{-1}, slightly shifted from the transformation of the sulfonamide (SO_2NH) to the enol form (SO(OH)N) as a result of complex formation giving a more stable six-membered ring. In the present system, IR data support that sulphonamide does not coordinate. In all of the metal complexes, there are new medium to weak bands appearing at frequencies between 548 and 560 cm^{-1}, assigned to ν(M–O) and bands at 412–475 cm^{-1} assigned to ν(M–N) [10]. Most oxovanadium(IV) complexes exhibit a strong band near 1000 cm^{-1}, assigned to ν(V=O). In contrast, several VO(IV) complexes have been reported in which this stretching mode appears at lower [18] wavenumbers, around 900 cm^{-1}. The shift of ν(V=O) to lower wavenumbers has been suggested due to the presence of a slight $\cdots V = O \cdots V = O \cdots$ interaction between the vanadyl oxygen of one molecule and a vanadium in another molecule [18, 19]. In this work, the ν(V=O) is found at 974 cm^{-1}, suggesting a slight $\cdots V = O \cdots V = O \cdots$ interaction. IR spectrum of the UO_2(VI) complex shows a strong absorption near 848 cm^{-1}, assigned to antisymmetric ν_3(O–U–O) [20]. IR bands of coordinated water at 825 cm^{-1} indicate the binding of water to the metal [12].

3.2. *Electronic spectra*

UV-Vis spectral absorptions of the ligand and Cu, Ni, Co, VO complexes were recorded in DMSO. The free ligand exhibits strong bands around 25,126 and 29,326 cm^{-1}, which are reasonably accounted for $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions. The electronic spectrum of the Co(II) complex gave bands at 9407, 15,625, and 17,241 cm^{-1}, assigned to ${}^4T_{1g}(F) \rightarrow {}^4T_{2g}(F)$ [ν_1], ${}^4T_{1g}(F) \rightarrow {}^4A_{2g}(F)$ [ν_2], and ${}^4T_{1g}(F) \rightarrow {}^4T_{2g}(F)$ [ν_3], respectively, suggesting octahedral geometry. The spectrum also shows a band at 25,000 cm^{-1} assigned to charge transfer. Nickel(II) complex also exhibits three bands in its electronic spectrum at 9790, 15,432, and 24,750 cm^{-1}, assigned to ${}^3A_{2g}(F) \rightarrow {}^3T_{2g}(F)$ [ν_1], ${}^3A_{2g}(F) \rightarrow {}^3T_{1g}(F)$ [ν_2], and ${}^3A_{2g}(F) \rightarrow {}^3T_{1g}(P)$ [ν_3], respectively, from octahedral geometry [21, 22]. The spectrum shows a band at 26,642 cm^{-1}, which may be attributed to ligand-to-metal charge transfer.

The octahedral geometry of Co(II) and Ni(II) complexes is further supported by the ν_2/ν_1 ratio of 1.6, lower than that of regular octahedral aqua complexes, due to the asymmetric environment around Co(II) and Ni(II). Ligand field parameters, ligand field splitting energy (10 Dq), Racah-interelectronic repulsion parameter (B), covalent factor (β), and ligand field stabilization energy (LFSE) have been calculated for the Ni(II) complex. The configuration interaction between high-spin $^3T_{1g}(P)$ and $^3T_{1g}(F)$ excited states gradually lowers the ratio of theoretical value from 1.8 to 1.6. The transition $^3A_{2g} \rightarrow {}^3T_{2g}$ corresponds to 10 Dq and with $15B' = \nu_2 + \nu_3 - 3\nu_1$, B' is 714 cm^{-1}. Taking the B value of nickel(II) as 1041 cm^{-1}, the interelectronic repulsion parameter $\beta = B'/B$ is 0.68. The B' value lower than the free ion value is an indication of orbital overlapping and delocalization of d-orbitals. The 45% covalency calculated using the formula $(B-B')/B \times 100$ reflects the extent of covalency in the M–L bonding. An approximate value of spin–orbit coupling constant λ has been obtained from $\lambda = 2.7 \times B^2/10\,D_q$ using the value of B. The μ_{eff} value calculated by substituting the experimental $10\,D_q$ and λ in $\mu_{\mathrm{eff}} = \mu_{\mathrm{s.o}}\ (1\text{–}4\,\lambda/\,10\,D_q)$ is in good agreement with the octahedral stereochemistry, which is 2.79 BM.

Visible spectra of Cu(II) complexes showing a broad band in the range 15,000–18,000 cm^{-1} may be interpreted in terms of square-planar stereochemistry, as $^2B_{1g} \rightarrow {}^2B_{2g}$ and $^2B_{1g} \rightarrow {}^2Eg$ transitions. The present Cu(II) complex exhibits a broad band around 14,045 and 17,700 cm^{-1}, and thus these are assigned to d–d transition suggesting square-planar geometry. Absence of any band below 10,000 cm^{-1} eliminates the possibility of tetrahedral or pseudo tetrahedral environment in these complexes [23].

Electronic absorption spectrum of oxovanadium complex in DMSO solution shows three d–d bands at 11,574, 18,761, and 24,210 cm^{-1} which are assigned as $^2B_2 \rightarrow {}^2E_1$, $^2B_2 \rightarrow {}^2B_1$, $^2B_2 \rightarrow {}^2A_1$ transitions, respectively [23, 24], consistent with the square-pyramidal geometry. An intense peak at 26,810 cm^{-1} is due to ligand–metal charge transfer. The complexes of Zn(II), Cd(II), and UO_2(VI) are diamagnetic. In analogy with those described for these complexes containing N–O donor Schiff bases and according to the empirical formula of these complexes, we proposed an octahedral geometry for Zn(II), Cd(II), and seven coordination for UO_2(VI) complexes [12].

3.3. ^{1}H-NMR spectra

^{1}H-NMR spectra of the Schiff base and its diamagnetic Zn(II) complex were recorded in DMSO-d_6. The ^{1}H-NMR spectrum of the ligand reveals the presence of –CH=N proton signal at 8.96 ppm (s, 1H, CH=N), 12.70 ppm (brs, 1H) due to phenolic OH, 10.23 ppm (s, 1H) due to SO_2NH [25, 26], and 6.51–7.01 ppm (m, Ph), 7.82 ppm (m, 3H) due to C(NH), (NH_2). The signal observed at 3.36 ppm is assigned to coordinated water [11]. All the other signals were unchanged, except phenolic OH at 12.7 ppm disappeared in the spectrum of Zn(II) complex indicating deprotonation upon complex formation.

3.4. Mass spectra

The mass spectrum of the ligand exhibits molecular ion peak at m/z 318 (45%), corresponding to its molecular formula ($C_{14}H_{14}N_4O_3S$). The base peak at m/z 224

(100%) is $C_7H_8N_4SO_2$. Other important peaks at *m*/*z* 260 (45%), 149 (52%), and 103 (52%) by cleavage in different positions in HL molecule are shown in "Supplementary material". The FAB mass spectra of VO(IV) and UO_2(VI) complexes show the molecular ion peaks, respectively, at *m*/*z* 701 and 903 values showing their composition to be ML_2 and $ML_2 \cdot H_2O$. The FAB mass spectrum of the zinc complex showed peaks of appreciable intensity at *m*/*z* values of 738, 718, and 701. The *m*/*z* value of 738 corresponds to [$ZnL_2 \cdot 2H_2O$], 718 to [$ZnL_2 \cdot H_2O$], and 701 to [ZnL_2] with a chelated ligand moiety.

3.5. *ESR spectra*

The spin Hamiltonian parameters of the Cu(II) complex are listed in table 2. The observed spectral parameters show anisotropic ESR spectra with $g_{\|} > g_{\perp}$ characteristic of square-planar geometry [27, 28]. The g_{iso} value is less than 2.3 indicating the covalent character of the metal–ligand bond. The square-planar geometry can also be supported by the fact that the unpaired electron lies predominantly in the $d_{x^2-y^2}$ orbital, as evident from the value of the exchange interaction term "*G*", estimated from [27] $G = g_{\|} - 2.0023/g_{\perp} - 2.0023$.

The α^2 value for the present complex of 0.86 indicates appreciable the in-plane covalency. The calculated value ($g_{\|}/A_{\|}$) 100.7 cm for the complex is an indication of its slightly distorted structure. The spin–orbit coupling constant (λ) for the copper ion in the complex is $-211.59\,\text{cm}^{-1}$, which is less than that of free Cu(II) ion ($=828\,\text{cm}^{-1}$), indicative of the covalency in Cu–L bonding [29–32].

The magnetic moment of the copper(II) complex calculated using the relation $\mu^2 = 3/4\ |g|^2$ and is 1.80 BM, indicative of an unpaired electron. The orbital reduction factors $K_{\|}$ and $K_{\perp}$ are estimated from the expression $K_{\|} = (g_{\|} - 2.0023)\ \Delta E/8\lambda$, $K_{\perp} = (g_{\|} - 2.0023)\ \Delta E/2\lambda$, $\lambda = -828\,\text{cm}^{-1}$ (spin–orbit coupling constant for the free ion). In case of a pure σ bonding $K_{\|} \cong K_{\perp}$, whereas $K_{\|} < K_{\perp}$ implies considerable in-plane π-bonding, while for out of π-plane bonding $K_{\|} > K_{\perp}$. For this complex, $K_{\|} > K_{\perp}$ indicates poor in-plane π-bonding which is also reflected in β^2 values [33].

3.6. *Redox behavior*

The redox behavior of the copper and oxovanadium complexes were studied using cyclic voltammetry at 10^{-3} M in DMSO (table 3). The oxovanadium [21, 27] complex exhibited two reduction peaks at 0.629 and 0.456 V and one oxidation peak at -0.0516 mV. The redox process involves V(IV) → V(III) $E_{\text{pc}} = 0.629$ V, V(III) → V(II)

Table 2. ESR spectral parameters.

Parameters	$g_{\|}$	$g_{\perp}$	g_{iso}	α^2	β^2	$A_{\|} \times 10^{-4}$ (cm^{-1})	$A_{\perp} \times 10^{-4}$ (cm^{-1})	$A_{\text{iso}} \times 10^{-4}$ (cm^{-1})	$g_{\|}/A_{\|}$ (cm)	$K_{\perp}$	μ (BM)	λ (cm^{-1})
CuL_2	2.2	2.02	2.05	0.86	0.25	218	54	109	100	0.12	1.80	–211

$E_{pc} = 0.456$ V, and V(II) → V(IV) $E_{pa} = -0.0516$ V. The cyclic voltammetry data for the complex with three different scan rates are listed in table 3.

E_{pc} and E_{pa} change with scan rate and ΔE_p values increase with increasing scan rate to more than 400 mV. These observations indicate that the electron transfer is irreversible. The CV data for the copper complex with four different scan rates are given in table 4. The cyclic voltammogram of the copper complex in DMSO at RT shows one reduction cathodic peak for Cu(II) → Cu(0) at $E_{pc} = 0.5136$ V. In the anodic side, the direct oxidation of Cu(0) → Cu(II) is observed with a strong stripping peak at $E_{pa} = -0.1125$ V [34]. E_{pc} and E_{pa} change with the scan rate and ΔE_p values increase with increasing the scan rate to more than 400 mV, indicating that the electron transfer is irreversible.

Table 3. Redox potential for the oxovanadium and copper complexes.

Compound	Scan rate (mV)	E_{pc} (mV)	E_{pa} (mV)	ΔE_p (mV)
VO(IV)	25	−0.632	0.0882	0.543
	50	0.481		
		0.690	−0.1655	0.524
		0.521		
Cu(II)	10	0.513	−0.1125	0.654
	30	0.543	−0.1737	0.716
	40	0.619	−0.2103	0.829
	50	0.643	−0.2181	0.862

Table 4. Antimicrobial activity of the Schiff base and its metal complexes.

		Zone of inhibition (in mm)[a]		
Compound	Concentration (mg mL^{-1})	*Escherichia coli*	*Staphylococcus aureus*	*Candida*
Sulfaguanidine	05	20	23	16
Sulfaguanidine	10	17	12	12
	15	16	11	10
	05	18	12	R
Ligand	10	R	10	R
	15	R	R	R
	05	18	R	15
VO(IV)	10	06	R	11
	15	06	R	10
	05	R	12	15
Cu(II)	10	R	12	11
	15	10	11	R
	05	R	R	21
Ni(II)	10	R	R	20
	15	R	R	18
	05	17	17	17
Standard	10	17	17	17
	15	17	17	17

[a] All values are the mean ($n = -3$) with a standard deviation of $<3\%$; R = resistance.

3.7. *Biological activity*

Antibacterial and antifungal tests were carried out using disc diffusion method at 5, 10, and 15 mg mL^{-1} in DMSO. Amikacin and ketoconazole were used as reference compounds for antibacterial and antifungal activities, respectively. *E. coli* (Gram negative bacteria), *S. aureus* (Gram negative bacteria), and *Candida* (fungus) were used as test organisms. The test was done using diffusion agar technique. Results in the form of inhibition zones (mm) are tabulated in table 4. The Schiff-base complexes exert higher antimicrobial activity than free ligand, especially Ni(II) complex and VO(IV) complexes were more effective on fungi than bacteria. Previous work [10] reveals that Ni(II) complexes show highest antimicrobial activities on Gram negative, Gram positive, and fungi. The greater activity can be explained on the basis of particle size and size of metal ion [10] and on chelation theory [35–38]. It has been suggested that the mode of action of the compounds may involve the formation of hydrogen bond through azomethine group with the active center of cell constituents resulting in interference with normal cell process [36].

4. Conclusion

The Schiff-base complexes are bidentate, coordinated through azomethine nitrogen and phenolic oxygen, as confirmed by analytical and spectral studies. Sulfonamide does not take part in bonding [14, 20]. The ligands have less ligand field strength than salicylaldimines producing high-spin octahedral complexes. Metal chelates show better antimicrobial activity than the free ligand.

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