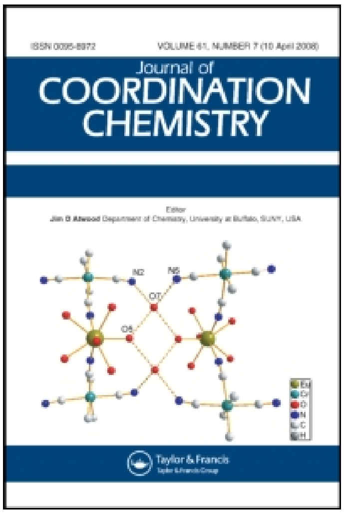

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Synthesis and spectral characterization of a new symmetric bidentate Schiff-base and its zinc complexes

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Synthesis and spectral characterization of a new symmetric bidentate Schiff-base and its zinc complexes

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A new symmetric bidentate Schiff base *N*,*N′*-*bis* [(E)-3-(2-nitrophenyl)allylidene)]benzene-1,2-diamine (L) and its complexes with general formula $ZnLX_2$ (X = chloride, bromide, iodide, thiocyanate and azide) are described. The authenticity of the ligand and complexes have been established by microanalysis, electronic, MS, FT-IR, ^1H and ^{13}C NMR spectra, and by molar conductivity measurements. All compounds are non-electrolytes in DMF. The analytical data confirmed that the metal to ligand ratio in the complexes is 1 : 1. The complexes have pseudotetrahedral geometry with C_{2V} point group.

Keywords: Schiff-base; Azide; Thiocyanate; Zinc(II) complexes; Spectral

1. Introduction

Metal complexes with Schiff-bases as ligands play an important part in development of coordination chemistry. The ability of different metal ions to arrange the coordinating ligands in different orientations is the reason for development of different assembly structures [1–4]. Transition metal complexes of Schiff-base ligands find applications as models for metal enzymes, catalysts and modifiers in materials chemistry [5]. Schiff-base metal complexes have been a widely studied subject because of their industrial, antifungal, biological applications and other interesting properties [6–9]. Schiff-bases and their biologically-active complexes have often been used as radiopharmaceuticals for cancer targeting, agrochemicals, as model systems for biological macromolecules, as catalysts and as dioxygen carriers [10–14]. Schiff-bases can be used to obtain optical materials and conducting polymers [15, 16]. Compounds containing Schiff-bases are suitable candidates for further chemical modifications and may be optical communication and optical devices [17].

The present study deals with synthesis and characterization of *N*,*N′*-*bis* [(E)-3-(2-nitrophenyl)allylidene)]benzene-1,2-diamine (L) as bidentate ligand and its metal complexes with general formula of $ZnLX_2$ (X = chloride, bromide, iodide, thiocyanate and azide). The complexes have pseudotetrahedral geometry in C_{2V} point group. Molar conductivity indicated that all complexes are mononuclear.

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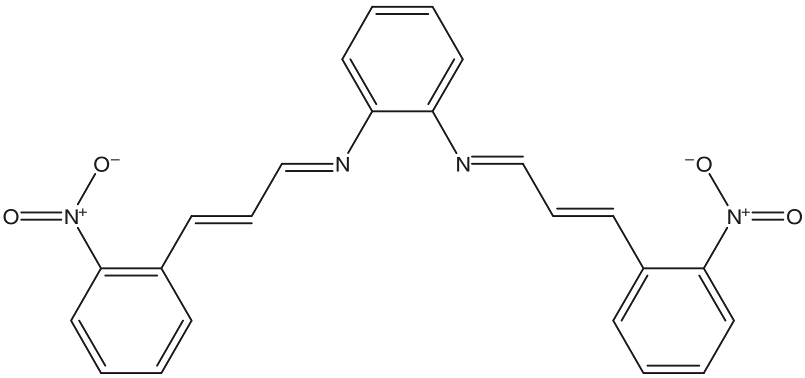

Figure 1. Structure of the Schiff-base.

2. Experimental

2.1. *Materials*

(E)-3-(2-nitrophenyl)-2-propenal, 1,2-benzenediamine, zinc salts and other chemicals were obtained from Aldrich and Merck. Solvents were purified by standard methods [18].

2.2. *Physical measurements*

Infrared spectra (4000–400 cm^{-1}) were recorded as KBr pellets on a FT-IR (JASCO-680 model) spectrophotometer. UV–Vis spectra were recorded using a JASCO-V570 model spectrophotometer in DMF. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker DPX-300 MHz FT-NMR spectrometer with the samples dissolved in DMSO-d_6 using TMS as internal standard. MS (*m*/*z*) of ligand was recorded on Shimadzu model GC-MS QP5050. Elemental analyses (CNHS) of dried samples were performed using a CHNS-932 (leco) elemental analyzer by central instrumental laboratory of Tarbiat Moallem University of Tehran. The melting points (°C) of the compounds were recorded on a BI Barnstead electrothermal instrument. The molar conductivity of the ligands and their complexes were determined in DMF (1.0×10^{-3} M) at room temperature using a Metrohm 712 conductometer with a dip-type conductivity cell made of platinum black.

2.3. *Synthesis of Schiff-base*

The new Schiff-base *N*,*N*′-*bis* [(E)-3-(2-nitrophenyl)allylidene)]benzene-1,2-diamine (L) was prepared by condensation of (E)-3-(2-nitrophenyl)-2-propenal (10 mmol, 1.77 g) with 1,2-benzenediamine (5 mmol, 0.54 g) in 60 mL methanol under reflux with rigorous stirring for 6 h. The Schiff-base precipitated on cooling, the reaction mixture was filtered, the ligand was separated and washed with a small amount of methanol. The compound was recrystallized from dichloromethane/ethanol, m.p.: 146–148°C (1.44 g, yield 68%). The Schiff-base is soluble in dichloromethane, chloroform, acetone, dimethylsulfoxide, dimethylformamide, and less soluble in alcohols. The structure of the Schiff-base is presented in figure 1.

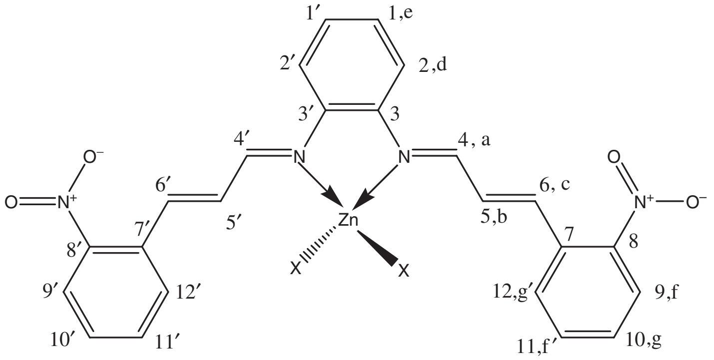

Figure 2. Structure of $ZnLX_2$ ($X = Cl^-$, Br^-, I^-, SCN^- and N_3^-). The letters and numbers refer to hydrogens and carbons of the structures, respectively (carbon atoms of thiocyanates are referred as $C_{(13,13')}$).

2.4. *Preparation of ZnX_2 (X = thiocyanate and azide)*

Solution of 0.5 mmol (0.149 g) zinc nitrate hexa hydrate in 15 mL ethanol was mixed with solution of 1 mmol of sodium azide (0.065 g) or potassium thiocyanate (0.097 g) in 15 mL methanol. The reaction mixture was stirred at room temperature for 2 h and then the white precipitate of sodium or potassium nitrate was separated and the filtrate containing zinc azide or zinc thiocyanate was used for complexation.

2.5. *Synthesis of $ZnLX_2$ (X = chloride, bromide, iodide, thiocyanate and azide)*

Solution of 0.5 mmol (0.213 g) ligand in 20 mL dichloromethane/methanol mixture was mixed with solution of 0.5 mmol of ZnX_2 ($ZnCl_2$ (0.068 g), $ZnBr_2$ (0.113 g) and ZnI_2 (0.159 g)) in methanol (30 mL) (except for zinc azide and zinc thiocyanate where 0.5 mmol of freshly prepared solution in 30 mL ethanol). The mixture was vigorously stirred at room temperature for 4–6 h, the resulting mixture was reduced to 1/3 volume by vacuum distillation, the reaction mixture was filtered and washed twice with warm methanol to give yellow to orange precipitates. The precipitates were further purified from DMF/ethanol mixture, dried at 80–100°C under vacuum and kept in a desiccator over silica gel. The complexes decompose at 185–318°C. The complexes are insoluble in common organic solvents such as dichloromethane, chloroform, acetone and acetonitrile, and are soluble in coordinating solvents such as DMSO and DMF. Structure of the complexes is shown in figure 2.

3. Results and discussion

3.1. *Physical data*

The reaction of ZnX_2 (X = chloride, bromide, iodide, thiocyanate and azide) with *N,N′-bis* [(E)-3-(2-nitrophenyl)allylidene)]benzene-1,2-diamine in a 1 : 1 (MX_2 : L) molar ratio gives complexes whose stoichiometries are confirmed by elemental analysis. Elemental analyses and other physical properties of the ligand and its complexes are

Table 1. Synthetic, analytical and conductivity data for the ligand and its zinc complexes.

					Found (Calcd) (%)			
Compound	Mol. wt. (Calcd)	Color	M.p. (°C)	Yield (%)	C	N	H	Λ_M ($cm^2\,\Omega^{-1}M^{-1}$)
L	426.42	Yellow	146–148	68	67.60 (66.85)	13.14 (13.20)	4.25 (4.22)	2.18
$ZnLCl_2$	562.72	Light orange	309 (dec.)	78	50.66 (51.23)	9.80 (9.96)	3.07 (3.22)	46.10
$ZnLBr_2$	651.62	Yellow	302 (dec.)	88	44.20 (44.24)	8.62 (8.60)	2.70 (2.78)	57.5
$ZnLI_2$	745.62	Orange	318 (dec.)	81	38.71 (38.66)	7.48 (7.51)	2.45 (2.43)	94.20
$ZnL(SCN)_2$	607.98	Orange	256 (dec.)	75	51.03 (51.36)	13.55 (13.82)	3.03 (2.98)	36.30
$ZnL(N_3)_2$	575.85	Yellow	185 (dec.)	65	51.32 (50.06)	22.93 (24.32)	3.04 (3.15)	31.02

Table 2. Infrared (cm^{-1}) and UV–Vis (nm) spectral data of the Schiff-base and its Zn(II) complexes.

Compound	–C=N	$–NO_2$	–N3	–SCN	M–N	λ (nm)	ε ($M^{-1}\,cm^{-1}$)
Ligand	1609(s)	1519(vs), 1347(vs)	–	–	–	374 278	14,686 43,122
$ZnLCl_2$	1618(s)	1522(vs), 1342(vs)	–	–	464(m)	374 280	12,331 38,261
$ZnLBr_2$	1615(s)	1523(vs), 1344(vs)	–	–	451(w)	376 282	19670 52,390
$ZnLI_2$	1613(s)	1523(vs), 1345(vs)	–	–	464(w)	374 280	21,205 60,286
$ZnL(N_3)_2$	1613(s)	1521(vs), 1343(vs)	2059(vs)	–	458(m)	398 282	10,665 42,582
$ZnL(SCN)_2$	1619(s)	1523(vs), 1350(vs)	–	2063(vs)	463(w)	372 282	13,526 41,335

summarized in table 1. The compounds are stable under ordinary laboratory conditions. Low molar conductance values of 10^{-3} M solutions of the complexes in DMF at room temperature, in the 31.02–94.20 $cm^2\,\Omega^{-1}M^{-1}$ range, show that they are all non-electrolytes [19, 20].

3.2. *IR spectra*

Characteristic infrared and electronic spectral data of the ligand and its zinc complexes are listed in table 2. The absence of functional group stretching frequencies of the reactants [1682 cm^{-1} for ν(–C=O) of the aldehyde and two absorption bonds at 3200–3300 cm^{-1} for ν_{sym} and ν_{asym} of ($–NH_2$) of 1,2-benzenediamine] indicate there is no starting material. The appearance of a new stretching frequency at 1609 cm^{-1}, in agreement with the expected structure of the ligand, can be attributed to stretching of –C=N. In the metal complexes, the stretching bond of the –C=N is shifted about 4–10 cm^{-1} to higher frequencies, at 1613–1619 cm^{-1}.

The observed shift in the –C=N stretching after complexation confirms coordination of the azomethine (–C=N) group to the metal ion. A literature survey [8, 19, 21] showing

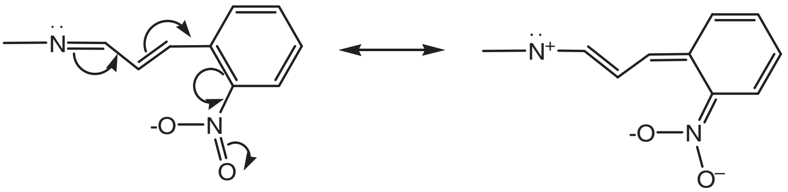

Figure 3. Probable resonance in the ligand.

stretching frequency of –C=N shifted to lower frequencies after coordination can be attributed to the π-back bonding of metal to ligand after σ-coordination of –C=N group. According to other reports, –C=N stretching in complexes shifted to higher frequencies can be explained based on figure 3 [1, 3b, 22, 23]. As shown in figure 3, because of the –NO_2 group in the ligand, resonance for the free ligand may be limited after coordination of –C=N. Therefore, the –C=N stretching frequency will shift to higher wavenumber. The very strong bands at 1519 and 1347 cm^{-1} for the ligand can be attributed to the asymmetric (ν_{asym}) and symmetric stretching (ν_{sym}) of –NO_2 groups. As shown in table 2, in the complexes these strong bands shift a few wavenumbers. Evidences for bonding in the complexes is also shown by observation of new bands in the spectra of the metal complexes at 451–464 cm^{-1} assigned to ν(M–N) [24]. The absorption at 2059 cm^{-1} in $ZnL(N_3)_2$ is assigned to coordinated N_3^- [25, 26] and the band at 2063 cm^{-1} in $ZnL(SCN)_2$ to coordinated SCN^- [25, 27].

3.3. *Electronic spectra*

Electronic spectra of the ligand and its complexes were recorded in DMF of 1.0×10^{-3} M at room temperature and the spectral data including the λ and ε are summarized in table 2. The spectrum of the free Schiff-base exhibits two absorption bands at 278 and 374 nm, attributed to π–π^* transitions, the first with $2\text{-}NO_2C_6H_4CH{=}CH$– and the second with imino. In the complexes, the imino-group π–π^* transition is shifted to longer wavelength as a consequence of coordination, confirming the formation of Schiff-base metal complexes [28]. The suggested structure for the zinc complexes [29] is pseudo-tetrahedral with C_{2v} point group (figure 2).

3.4. *Mass spectra*

Mass spectral data of the Schiff-base (Supplementary Material) includes main fragments of 424(M^+-2), 312, 270, 252, 235, 233, 219, 203, 168, 146, 117, 107, 85, 81, 79, 72, 57, 51(m/z). Electron impact (EI) mass spectroscopy (MS) lends support to the formula. A proposed fragmentation of the ligand is shown in figure 4. The mass spectrum of the ligand presents the peak corresponding to the molecular ion (M^+-2) that indicates stability of ligand in the gas phase. Other fragments are in agreement with the molecular formula of the ligand. The mass spectrum of $ZnLCl_2$ was recorded but its molecular ion was not observed and the fragmentation similar to the ligand was obtained.

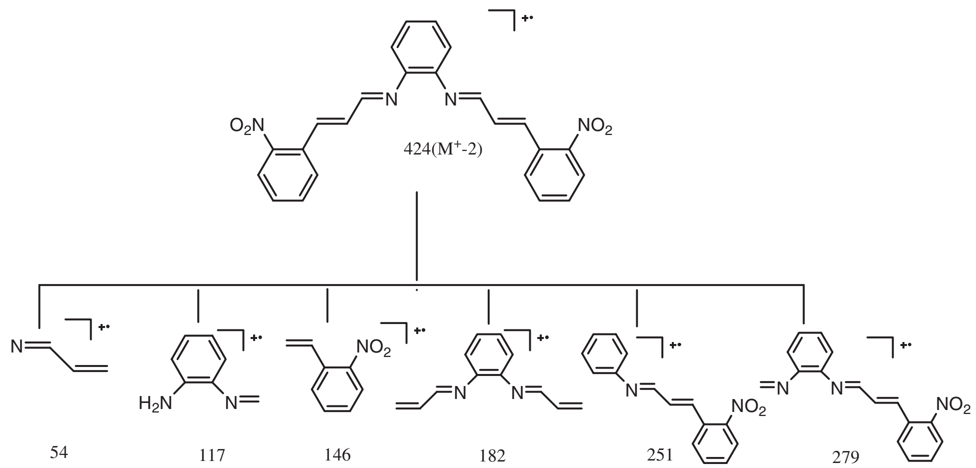

Figure 4. Some proposed fragments of the ligand based on mass spectrum.

Table 3. ^1H NMR chemical shifts of the ligand and its Zn(II) complexes in ppm in DMSO-d_6 solvent.

Compounds	Assignment of the protons
Ligand	8.33(d, $2H_a$, $J=8.80$ Hz), 8.03(d, $2H_f$, $J=7.46$ Hz), 8.01(d, $2H'_g$, $J=8.46$ Hz), 7.75(t, $2H'_f$, $J=7.46$ Hz and $J=7.56$ Hz), 7.61(d, $2H_c$, $J=15.93$ Hz), 7.59(t, $2H_g$, $J=5.83$ Hz and $J=10.10$ Hz), 7.14(dd, $2H_b$, $J=15.87$ Hz and $J=8.90$ Hz), 7.23(dd, $2H_e$, $J=5.78$ Hz and $J=3.45$ Hz) and 7.10(dd, $2H_d$, $J=5.74$ Hz and $J=3.51$ Hz) (Supplementary Material).
$ZnLCl_2$	8.50(d, $2H_a$, $J=8.09$Hz), 8.03(dd, $2H_f$, $J=8.09$ Hz and $J=1.00$ Hz), 7.97(d, $2H'_g$, $J=7.28$ Hz), 7.78(t, $2H'_f$, $J=6.92$ Hz and $J=7.61$ Hz), 7.69(d, $2H_c$, $J=15.78$ Hz), 7.63(t, $2H_g$, $J=7.74$ Hz and $J=7.84$ Hz) and 7.27(m, $6H_{bde}$) with detailing of {7.27(dd, $2H_b$, $J=15.82$ Hz and $J=8.88$ Hz) and 7.29(m, $4H_{e,d}$)}.
$ZnLBr_2$	8.51(d, $2H_a$, $J=8.88$ Hz), 8.03(dd, $2H_f$, $J=8.08$ Hz and $J=0.85$ Hz), 7.98(d, $2H'_g$, $J=7.35$ Hz), 7.78(t, $2H_{f'}$, $J=7.26$ Hz and $J=7.62$ Hz), 7.69(d, $2H_c$, $J=15.77$ Hz), 7.63(t, $2H_g$, $J=7.85$ Hz and $J=7.75$ Hz) and 7.27(m, $6H_{bde}$) with detailing of {7.28(dd, $2H_b$, $J=15.76$ Hz and $J=8.73$ Hz), 7.27(bs, $4H_{e,d}$)}.
$ZnLI_2$	8.60(d, $2H_a$, $J=8.86$ Hz), 8.04(d, $2H_f$, $J=8.07$ Hz), 7.93(d, $2H'_g$, $J=7.65$ Hz), 7.81(t, $2H'_f$, $J=7.50$ Hz and $J=8.92$ Hz), 7.75(d, $2H_c$, $J=16.15$ Hz), 7.65(t, $2H_g$, $J=8.00$ Hz and $J=7.46$ Hz) and 7.36(m, $6H_{bde}$) with detailing of {7.35(dd, $2H_b$, $J=15.91$ Hz and $J=8.4$ Hz), 7.34(bs, $4H_{e,d}$)}.
$ZnL(SCN)_2$	8.56(d, $2H_a$, $J=9.02$ Hz), 8.04(t, $4H_{f,g'}$, $J=8.67$ Hz and $J=8.59$ Hz), 7.79(t, $2H'_f$, $J=7.11$ Hz and $J=7.81$ Hz), 7.77(d, $2H_c$, $J=15.57$ Hz), 7.66(t, $2H_g$, $J=7.90$ Hz and $J=7.66$ Hz), 7.48(dd, $2H_b$, $J=15.66$ Hz and $J=9.03$ Hz) and 7.34(m, $4H_{e,d}$).
$ZnL(N_3)_2$	8.54(d, $2H_a$, $J=8.97$ Hz), 8.04(dd, $2H_f$, $J=8.10$ Hz and $J=0.74$ Hz), 7.99(d, $2H'_g$, $J=7.61$ Hz), 7.78(t, $2H'_f$, $J=7.40$ Hz and $J=8.28$ Hz), 7.73(d, $2H_c$, $J=15.80$ Hz), 7.65(t, $2H_g$, $J=7.64$ Hz and $J=7.37$ Hz), 7.39(dd, $2H_b$, $J=15.65$ Hz and $J=8.95$ Hz) and 7.31(m, $4H_{e,d}$) with detailing of {7.33 (dd, $2H_e$, $J=5.96$ Hz and $J=3.30$ Hz), 7.28(dd, $2H_d$, $J=5.98$ Hz and $J=3.34$ Hz)}.

3.5. *^1H NMR and ^{13}C NMR spectra*

The NMR spectra of the ligand and its zinc complexes were recorded using DMSO-d_6 at 300 MHz. The ^1H and ^{13}C NMR spectral studies of the ligand (Supplementary Material)

Table 4. ^{13}C NMR chemical shifts of the ligand and its Zn(II) complexes in ppm in DMSO-d_6 solvent.

Compounds	Assignment of carbon atoms												
	$C_{(1,1')}$	$C_{(2,2')}$	$C_{(3,3')}$	$C_{(4,4')}$	$C_{(5,5')}$	$C_{(6,6')}$	$C_{(7,7')}$	$C_{(8,8')}$	$C_{(9,9')}$	$C_{(10,10')}$	$C_{(11,11')}$	$C_{(12,12')}$	$C_{(13,13')}$
Ligand	120.21	124.53	148.109	144.36	137.62	128.56	126.58	162.74	137.62	132.80	133.57	130.18	–
$ZnLCl_2$	119.96	124.64	148.12	143.43	139.12	128.49	127.33	163.34	139.12	132.18	133.70	130.49	–
$ZnLBr_2$	119.98	124.65	148.12	143.42	139.12	128.49	127.32	163.32	139.12	132.17	133.71	130.50	–
$ZnLI_2$	119.92	124.73	148.11	142.97	140.07	128.41	127.80	163.84	140.07	131.75	133.83	130.72	–
$ZnL(SCN)_2$	119.72	124.74	148.16	143.01	140.27	128.53	127.92	164.09	140.27	131.52	133.73	130.73	134.91
$ZnL(N_3)_2$	119.79	124.69	148.19	143.21	139.70	128.46	127.69	163.75	139.70	131.79	133.70	130.62	–

and its zinc complexes provide clues to the geometry. For example, the ^{1}H NMR spectrum of the ligand shows the azomethine proton (–CH=N) resonance at 8.33 ppm, which shifts to 8.50–8.60 ppm in its zinc(II) complexes, suggesting coordination by azomethine nitrogen. Ethylenic protons H_b and H_c (with *trans*-position around of –C=C–) are at 7.61(d, 2H_c, $J = 15.93$ Hz) and 7.14(dd, 2H_b, $J = 15.87$ Hz and $J = 8.90$ Hz). Coupling between these protons of 15–16 Hz supports our structure [30]. Similarly, ethylenic protons and others in the complexes are assigned. The ^{13}C NMR spectrum of the ligand shows the azomethine carbon (–CH=N) at 137.62 ppm, which shifts to 139.12–140.27 ppm in the complexes, confirming coordination of azomethine nitrogen. The proposed assignments of ^{1}H and ^{13}C NMR spectral data are summarized in tables 3 and 4 for the labels in figure 2.

Acknowledgement

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