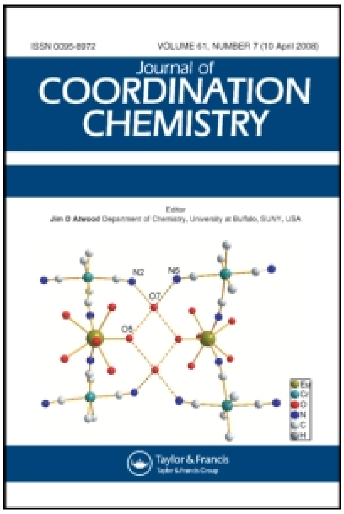

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Synthesis and spectroscopic studies of a new unsymmetrical ligand, <i>N</i>(<i>E</i>),<i>N'</i>(<i>E</i>)-<i>bis</i>[(<i>E</i>)-2-methyl-3-phenylallylidene]propane-1,2-diamine, and its zinc complexes

Morteza Montazerozohori[a]; Shiva Joohari[a]; Sayed Alireza Musavi[a]
[a] Department of Chemistry, Yasouj University, Yasouj, Iran

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Synthesis and spectroscopic studies of a new unsymmetrical ligand, *N*(*E*),*N*′(*E*)-*bis*[(*E*)-2-methyl-3-phenylallylidene] propane-1,2-diamine, and its zinc complexes

MORTEZA MONTAZEROZOHORI*, SHIVA JOOHARI and SAYED ALIREZA MUSAVI

Department of Chemistry, Yasouj University, Yasouj, Iran

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A new unsymmetrical bidentate Schiff base *N*(*E*),*N*′(*E*)-*bis*[(*E*)-2-methyl-3-phenylallylidene]-propane-1,2-diamine and four zinc complexes with general formula Zn(BMPAPD)X_2 in which $X = Cl^-$, Br^-, I^-, or SCN^-, have been prepared and characterized by elemental analysis, mass spectrometry, ^{1}H NMR (Nuclear Magnetic Resonance), ^{13}C NMR, Fourier Transform–Infrared and Ultraviolet–Visible spectra, and molar conductance. All compounds are nonelectrolytes in dimethyformamide. The molar conductance and spectral properties indicate coordination of halides and thiocyanate ions to zinc in pseudo-tetrahedral geometry.

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

1. Introduction

Schiff bases (azomethines) are widely used as biologically active substances, liquid crystals, dyes, luminophores and polymer stabilizers [1–3]. New applications such as antidepressants, antimicrobial, antitumor, antiphlogogistic, nematocide, and other medicinal agents have been reported for these compounds [4, 5]. A wide range of transition metal complexes derived from Schiff-base ligands have been isolated [6–11]. The field of Schiff-base complexes is fast developing on account of the wide variety of possible structures for the ligands depending upon the aldehydes and amines and the interesting properties [12–14]. Schiff bases and their biologically active complexes have also been used as radiopharmaceuticals for cancer targeting, agrochemicals, as model systems for biological macromolecules, as catalysts and as dioxygen carriers [15–19]. Four-coordinate zinc complexes of bidentate Schiff-base ligands are rare.

In continuation of our studies on four-coordinate complexes [20–23], we report the synthesis, physical, and spectral characterization of a new unsymmetrical bidentate Schiff base, *N*(*E*),*N*′(*E*)-*bis*[(*E*)-2-methyl-3-phenylallylidene]propane-1,2-diamine (BMPAPD), and its zinc complexes of general formula Zn(BMPAPD)X_2 ($X = Cl^-$, Br^-, I^-, and SCN^-).

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*Corresponding author. Email: mmzohori@mail.yu.ac.ir; mmzohory@yahoo.com

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2. Experimental

2.1. *Materials*

(*E*)-2-methyl-3-phenyl-2-propenal, 1,2-propanediamine, zinc salts, and other chemicals were purchased from either Aldrich, Merck or BDH Chemicals and used without purification.

2.2. *Physical measurements*

Infrared (IR) spectra (KBr pellets) were recorded on a JASCO-680 model FT–IR spectrometer in the 4000–400 cm^{-1} range. UV–Visible (UV–Vis) spectra were recorded in chloroform on a JASCO-V570 model spectrometer. ^1H and ^{13}C NMR (Nuclear Magnetic Resonance) spectra were obtained using a Bruker DPX FT–NMR spectrometer at 500 MHz with the samples dissolved in $CDCl_3$ or $CDCl_3$/DMSO (Dimethyl Sulfoxide-d_6) mixture using trimethysilyl as internal standard. Mass spectrometry (MS; m/z) of the ligand was recorded on Shimadzu model GC–MS QP5050. Elemental analyses (CNHS) of dried samples were performed using a CHNS elemental analyzer of the central instrumental laboratory of Tarbiat Moallem University of Tehran. The melting points (°C) of the complexes were recorded on a BI Barnstead electrothermal instrument. The molar conductivities of the ligands and their complexes were determined in dimethyformamide (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 ligand BMPAPD*

A solution of (*E*)-2-methyl-3-phenyl-2-propenal (0.584 g, 4 mmol) in absolute methanol (10 mL) was added to a solution of 1,2-propanediamine (0.15 g, 2 mmol) in absolute methanol (10 mL) and vigorously stirred for 3–4 h. After completion of the reaction, the solvent was reduced under vacuum to give the Schiff base as an yellowish-white viscous oil. For purification, the oil was washed twice with *n*-hexane and/or *n*-heptane and dried under vacuum to obtain the ligand with good purity in 72% yield (0.48 g). The Schiff base is soluble in dichloromethane, chloroform, acetone, dimethylsulfoxide, dimethylformamide, less soluble in alcohols, and not soluble in *n*-hexane and *n*-heptane.

2.4. *Preparation of Zn(BMPAPD)X_2 ($X = Cl^-$, Br^-, I^-, and SCN^-) complexes*

The zinc(II) complexes were prepared by addition with stirring of the ligand (0.165 g, 0.5 mmol) in methanol (10 mL) to 0.5 mmol of zinc halide ($ZnCl_2$(0.068 g), $ZnBr_2$(0.113 g), ZnI_2(0.159 g), and $Zn(SCN)_2$ freshly prepared in ethanolic solution [23]) in methanol (30 mL) for 1–3 h. The white complexes, Zn(BMPAPD)X_2, so obtained were filtered, washed with ethanol twice and dried under vacuum (table 1). All of the isolated complexes are soluble in most organic solvents. The precipitates were further purified from dichloromethane/ethanol mixture, dried at (80–100°C) under vacuum and kept in a desiccator over silica gel. The melting points of the complexes were 197–208°C. The proposed structure of the ligand and complexes is shown in figure 1.

Table 1. Some analytical data and physical properties of the ligand and its zinc complexes.

Compound	Mol. wt. (Calcd)	Color	M.P. (°C)	Yield (%)	Found (Calcd) (%)			Λ_M (cm^2 Ω^{-1} M^{-1})
					C	H	N	
BMPAPD (**1**)	330.47	Light yellow	Viscous oil	72	83.75 (83.59)	7.71 (7.93)	8.32 (8.48)	2.08
Zn(BMPAPD)Cl_2 (**2**)	466.76	White	208	79	59.51 (59.18)	5.49 (5.61)	5.91 (6.00)	16.29
Zn(BMPAPD)Br_2 (**3**)	555.66	White	206	69	49.31 (49.18)	4.59 (4.72)	5.21 (5.04)	19.60
Zn(BMPAPD)I_2 (**4**)	649.66	Yellowish-white	202	75	42.31 (42.50)	4.29 (4.03)	4.21 (4.31)	67.17
Zn(BMPAPD)$(SCN)_2$ (**5**)	512.02	White	197	65	58.41 (58.64)	5.25 (5.12)	10.71 (10.94)	42.07

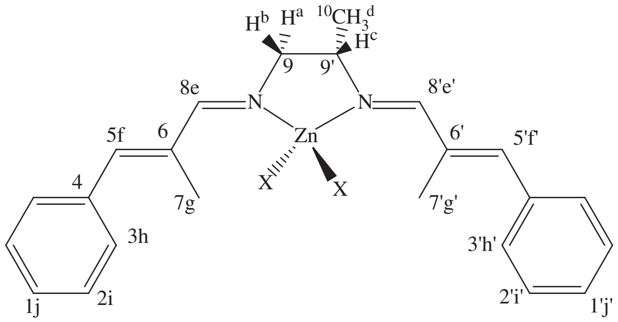

Figure 1. The structure of Zn(BMPAPD)X_2 ($X = Cl^-$, Br^-, I^-, and SCN^-) with labeled hydrogens and carbons.

3. Results and discussion

3.1. *Physical data*

Elemental analyses show 1 : 1 stoichiometry of ZnX_2 : BMPAPD for the complexes. The analytical data and physical properties of the complexes are summarized in tables 1 and 2. The low molar conductivities of 10^{-3} M solutions in DMF at room temperature of 16.29–67.17 $cm^2\,\Omega^{-1}\,M^{-1}$ show that they are all nonelectrolytes with the anions coordinated to the metal ion in a pseudo-tetrahedral geometry [24, 25].

3.2. *IR spectral investigation*

Table 2 presents the characteristic vibrational frequencies of BMPAPD and the zinc complexes. Frequencies assigned to the starting material including aldehyde and 1,2-propanediamine at 1680 and 3100–3300 cm^{-1} were not observed in the spectrum of the ligand. The FT–IR spectrum of BMPAPD showed frequencies at 3054, 3024, (2964, 2925) and 2858 cm^{-1} assigned to C–H of aromatic, olefinic, aliphatic, and iminic groups, respectively, not affected by coordination of the ligand. The vibrational frequency at 1626 cm^{-1} can be assigned to azomethine group (C=N) [6–10] and shifts by 18–20 cm^{-1} to lower frequencies in the spectra of the complexes, indicating coordination through the azomethine nitrogen (figure 1) [8, 24, 26]. The band at 1358 cm^{-1} present in the IR spectrum of the Schiff base may be assigned to (C–N) stretching mode that is shifted to 1348 cm^{-1} after coordination. The frequencies at 1490 and 1447 cm^{-1} show C=C stretching vibrations. The bending vibration of methyl groups is at 1385 cm^{-1}. The very strong out-of-plane bending of the aromatic C-H and C-C are present at 752 and 698 cm^{-1} [27], nearly unchanged after coordination. In the complexes, bands at 440–472 and 437–438 cm^{-1}, absent in the ligand spectrum, may be due to symmetrical and unsymmetrical vibration of (M–N) [18], suggesting that the complexes are four-coordinate with two azomethine nitrogens of bidentate ligand and two halides. The new intense band at 2072 cm^{-1} in the IR spectrum of Zn(BMPAPD)$(SCN)_2$ is safely attributed to *N*-coordinated SCN^- [7].

Table 2. FT–IR (cm^{-1}) and UV–Vis (nm) spectral data of the Schiff base BMPAPD and its zinc complexes.

Compound	$\nu CH_{arom.}$	νCH_{alkene}	$\nu CH_{aliph.}$	$\nu CH_{imin.}$	$\nu C{=}N$	$\nu C–N$	$\nu C{=}C$	δCH_{methyl}	$\delta CH_{arom(oop)}$ $\delta CC_{arom(oop)}$	$\nu Zn–X$ ($X = N$ and/or halide)	νSCN	λ_{max} (Є, cm^{-1} M^{-1})
1	3054(w)	3024(w)	2964(m), 2925(m)	2858(m)	1626(vs)	1358(m)	1490(m), 1447(m)	1395(w)	752(s), 698(s)	–	–	276(34980),282, 290
2	3048(w)	3023(w)	2973(w), 2921(w)	2864(w)	1608(vs)	1348(s)	1489(w), 1443(s), 1417(m)	1370(m)	754(s), 699(s)	472(m), 455(w), 438(w)	–	276(50258),280, 290
3	3051(w)	3023(w)	2969(w), 2913(w)	2864(w)	1606(vs)	1348(m)	1489(w), 1439(s)	1369(m)	753(s), 697(s)	470(m), 448(w), 437(w)	–	276(46708),280, 290
4	3046(w)	3023(w)	2970(w), 2925(w)	2869(w)	1606(vs)	1348(m)	1489(w), 1439(s)	1368(w)	753(s), 698(s)	470(m), 440(w)	–	274(55133),282, 290
5	3053(w)	3026(w)	2966(w), 2923(w)	2867(w)	1607(vs)	1348(s)	1441(s), 1420(sh)	1368(m)	754(s), 698(s)	472(m), 444(m), 437(w)	2072	276(51186),282, 292

Table 3. ^{1}H NMR chemical shifts of ligand and its zinc complexes in parts per million in $CDCl_3$ as solvent.

Compound	Assignment of the protons
1	8.02 (s, 1H_e), 7.97 (s, 1$H_{e'}$), 7.40 [m, 8H(2H_h, 2$H_{h'}$, 2H_i, 2$H_{i'}$)], 7.32 (t, 2$H_{jj'}$, $J = 5.80$ and 5.15 Hz), 6.81 (s, 1H_f), 6.80 (s, 1$H_{f'}$), 3.86 (dd, 1H_a, $J = 10.56$ and 4.63 Hz), 3.69 (sextet, 1H_c, $J = 6.10$ Hz), 3.66 (dd, 1H_b, $J = 10.59$ and 7.33 Hz) 2.18 (s, 3H_g), 2.17 (s, 3$H_{g'}$) and 1.36 (d, 3H_d, $J = 6.14$ Hz).
2	8.26 (s, 1H_e), 8.23 (d, 1$H_{e'}$, $J = 1.46$ Hz), 7.47 [m, 8H(2H_h, 2$H_{h'}$, 2H_i, 2$H_{i'}$)], 7.43 (m, 2$H_{jj'}$), 7.20 (s, 1H_f),7.19 (s, 1$H_{f'}$), 4.29 (bd, 1H_a, $J = 12.97$ Hz), 4.07 (m, 1H_c), 3.64 (dd, 1H_b, $J = 13.30$ and 1.18 Hz), 2.66 (d, 3H_g, $J = 1.07$ Hz), 2.65 (d, 3$H_{g'}$, $J = 1.18$ Hz) and 1.46 (d, 3H_d, $J = 6.84$ Hz).
3	8.26 (s, 1H_e), 8.22 (s, 1He′), 7.48 [m, 8H(2H_h, 2$H_{h'}$, 2H_i, 2$H_{i'}$)], 7.42 (m, 2$H_{jj'}$), 7.22 (s, 1Hf), 7.21 (s, 1$H_{f'}$), 4.32 (bd, 1H_a, $J = 12.98$ Hz), 4.08 (m, 1H_c), 3.62 (dd, 1H_b, $J = 13.36$ and 1.34 Hz), 2.71 (s, 3H_g), 2.65 (d, 3$H_{g'}$, $J = 0.86$ Hz) and 1.49 (d, 3H_d, $J = 6.86$ Hz).
4	8.26 (s, 1H_e), 8.22 (d, 1$H_{e'}$, $J = 1.16$ Hz), 7.50 (t, 4H (2H_h, 2$H_{h'}$) $J = 7.04$ and 6.10 Hz), 7.46 [dt, 4H(2H_i, 2$H_{i'}$), $J = 7.76$, 7.22 and 1.66 Hz], 7.42 (dt, 2H_{jj}, $J = 7.43$ Hz, $J = 6.84$ and 1.69 Hz), 7.25 (bs, 2$H_{ff'}$), 4.37 (bd, 1H_a, $J = 12.68$ Hz), 4.12 (m, 1H_c), 3.59 (dd, 1H_b, $J = 13.32$ and 1.46 Hz), 2.76 (d, 3H_g, $J = 1.07$), 2.65 (s, 3$H_{g'}$) and 1.54 (d, 3H_d, $J = 6.87$ Hz).
5[a]	8.13 (s, 1H_e), 8.11 (s, 1$H_{e'}$), 7.30 [m, 8H(2H_h, 2$H_{h'}$, 2H_i, 2$H_{i'}$)], 7.25 (m, 2$H_{jj'}$), 7.06 (bs, 2$H_{ff'}$), 3.88 (bd, 1H_a, $J = 12.70$ Hz), 3.86 (m, 1H_c), 3.51 (dd, 1H_b, $J = 12.80$ and 2.12 Hz), 2.19 (s, 3H_g), 2.18 (s, 3$H_{g'}$) and 1.18 (d, 3H_d, $J = 6.60$ Hz).

Note: [a]The ^{1}H NMR and ^{13}C NMR of **5** were recorded in $CDCl_3$/DMSO mixture.

3.3. *Electronic spectra investigation*

Electronic absorption data measured in DMF are given in table 2. In the spectrum of the ligand, three intense bands at 276, 282 (shoulder) and 290 (shoulder) nm shift to lower or higher wavelengths upon formation of the complexes, suggesting that the nitrogen of azomethine is coordinated to the metal. The values at 276 and 280 (shoulder) nm are attributed to the π–π* transition of the ethylenic and aromatic rings of the (PhCH=CH(Me)–) moieties. The shoulder at 290 nm is due to the π–π* transitions of the azomethine, localized within the imine chromophore. The electronic spectra of zinc complexes consist of charge transfer (L-M) transition that, in our complexes, is observed as a shoulder at 280 nm (overlapped with π–π* transition of the ethylenic moiety) [7]. The suggested structure for the zinc complexes, considering our previous report on this type of ligand [21], is pseudo-tetrahedral as drawn in figure 1.

3.4. *Mass spectra investigation*

Mass spectral data of the Schiff base (Supplementary Material) includes fragments of 329(M^+-1), 312, 268, 252, 235, 234, 217, 195, 187, 176, 173, 172, 158, 151, 145, 129, 118, 115, 103, 97, 83, 81, 79, 68, 64, 51 (m/z), lending support to the formula shown in figure 1. A probable fragmentation diagram of the ligand is proposed in Supplementary Material.

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

The NMR spectra of the ligand and complexes were recorded using $CDCl_3$ and/or $CDCl_3$/DMSO mixture as solvent at 500 MHz (tables 3 and 4). The ^{1}H and ^{13}C NMR spectra (Supplementary Materials) strongly support the geometry. The ^{1}H NMR spectrum of the ligand includes the azomethine (H_e and $H_{e'}$) resonances at 8.02–7.97 ppm [7]. These signals are red shifted to 8.13–8.26 ppm in the zinc(II)

Table 4. ^{13}C NMR chemical shifts of the ligand and its zinc complexes in parts per millian in CDCl$_3$ solvent.

Compounds	Assignment of carbon atoms										
	$\mathrm{C}_{(1',1)}$	$\mathrm{C}_{(2',2)}$	$\mathrm{C}_{(3',3)}$	$\mathrm{C}_{(4',4)}$	$\mathrm{C}_{(5',5)}$	$\mathrm{C}_{(6',6)}$	$\mathrm{C}_{(7',7)}$	$\mathrm{C}_{(8',8)}$	$\mathrm{C}_{(9',9)}$	$\mathrm{C}_{(10)}$	$\mathrm{C}_{(\mathrm{SCN})}$
1	137.10	137.14	129.79, 128.72	137.36, 137.27	139.48, 139.23	128.10, 128.01	13.85, 13.68	167.94, 165.95	68.10, 66.68	20.96	–
2	130.42	134.80	130.39, 129.82	135.45, 134.88	149.11, 149.04	129.80, 129.04	15.25, 15.18	175.30, 173.70	65.61, 65.17	21.01	–
3	130.47	134.70	130.43, 129.85	135.45, 134.79	149.23, 149.13	129.82, 129.04	16.46, 16.36	175.34, 173.87	65.60, 65.46	21.13	–
4	130.55	134.45	130.50, 129.88	135.45, 134.60	149.18, 149.01	129.85, 129.03	18.34, 18.19	175.26, 173.98	65.90, 65.36	21.34	–
5[a]	135.29	135.32	130.23, 130.21	138.32, 138.27	–	–	13.59, 13.57	–	65.06, 64.75	20.69	135.29

Note: [a]The ^{1}H NMR and ^{13}C NMR of **5** were recorded in CDCl$_3$/DMSO mixture.

complexes, suggesting coordination of the azomethine nitrogens to zinc. The olefinic protons of the ligands (H$_f$ and H$_{f'}$) at 6.81(s, 1H$_f$), 6.80(s, 1H$_{f'}$) shift to weaker fields ($>$7.06 ppm) in the complexes. These protons in **1**, **2**, and **3** are separate and uncoupled with any other protons, while they appear together in **4** and **5**.

In the spectrum of the ligand, H$_a$ is at 3.86 ppm as a doublet of doublets due to coupling with the H$_c$ and H$_b$, respectively. H$_b$ is similar to H$_a$ at 3.66 ppm as a doublet of doublets due to couplings with H$_a$ and H$_c$, respectively. In all complexes H$_a$ is a broad doublet but H$_b$ stayed a doublet of doublets and the signals of both protons shift to weaker fields after coordination. H$_c$ in the ligand spectrum is a clear sextet because of its coupling with H$_d$ and then with H$_a$ and H$_b$ with coupling constant of 6.10 Hz, but is a multiplet in all complexes. H$_d$ of the ligand is a doublet at 1.36 ppm due to coupling with H$_c$ and shifts to 1.46–1.54 ppm in **2**, **3**, and **4** and 1.18 ppm in **5**. H$_g$ and H$_{g'}$ of the ligand at 2.17–2.18 ppm are at weaker fields in **2**, **3**, and **4** but at stronger field for **5**. These observations for H$_d$, H$_g$, and H$_{g'}$ may be related to more effective π-back bonding from metal to ligand in **2**, **3**, and **4** with respect to **5**. Aromatic protons of the ligand and complexes are two multiplets except for **4**, as shown in table 3. The ^{13}C NMR spectrum of the ligand shows two azomethine carbons $\mathrm{C}_{(8',8)}$ resonances at 167.94 and 165.95 ppm because of the unsymmetrical ligand. These peaks red shift to 173.70–175.34 ppm in the zinc(II) complexes, suggesting coordination of the azomethine. The carbon of *N*-coordinated SCN$-$ appeared at 135.29 ppm. Among the other signals, $\mathrm{C}_{(3',3)}$, $\mathrm{C}_{(6',6)}$, $\mathrm{C}_{(7',7)}$, $\mathrm{C}_{(5',5)}$ and $\mathrm{C}_{(10)}$ are red shifted but $\mathrm{C}_{(1',1)}$, $\mathrm{C}_{(2',2)}$, $\mathrm{C}_{(4',4)}$, and $\mathrm{C}_{(9',9)}$ blue shift after coordination. Some carbons of **5** do not appear due to low solubility. For detailed assignment of the ^{1}H NMR and ^{13}C NMR peaks, the protons and the carbon atoms of the ligand and complexes are labeled as small letters and numbers in figure 1.

4. Conclusion

In this article, we report the synthesis and spectroscopic characterization of a new unsymmetrical bidentate Schiff-base ligand and its zinc complexes. The physical and spectral data suggest coordination of the ligand and halides/thiocyanate in pseudo-tetrahedral geometry. Due to the unsymmetrical structure of the ligand and complexes, these complexes may display different properties such as catalytic role, optical activity, etc. compared with similar complexes with symmetrical ligands.

Supplementary Materials

All of the MS, ^{1}H NMR and ^{13}C NMR spectra are available from authors and journal.

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