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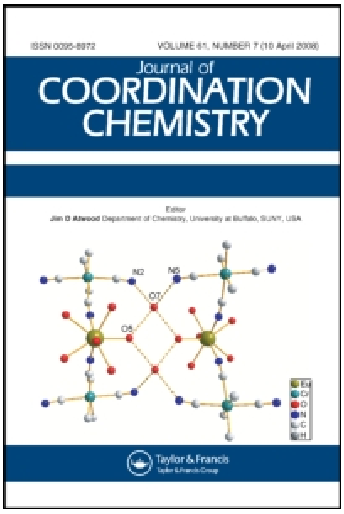

Journal of Coordination Chemistry

Publication details, including instructions for authors and subscription information:
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To cite this Article Canpolat, Erdal and Kaya, Mehmet(2005) 'Studies on mononuclear chelates derived from substituted Schiff-base ligands (part 7): synthesis and characterization of a new naphthyliden-*p*-aminoacetophenoneoxime and its complexes with Co(II), Ni(II), Cu(II) and Zn(II)', Journal of Coordination Chemistry, 58: 12, 1063 — 1069

To link to this Article: DOI: 10.1080/00958970500122565

URL: http://dx.doi.org/10.1080/00958970500122565

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Journal of Coordination Chemistry
Vol. 58, No. 12, 15 August 2005, 1063–1069

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Studies on mononuclear chelates derived from substituted Schiff-base ligands (part 7): synthesis and characterization of a new naphthyliden-*p*-aminoacetophenoneoxime and its complexes with Co(II), Ni(II), Cu(II) and Zn(II)

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(*Received 28 June 2004; revised 28 September 2004; in final form 31 March 2005*)

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In this study, the new ligand, naphthyliden-*p*-aminoacetophenoneoxime (LH) was synthesized starting from *p*-aminoacetophenoneoxime and 2-hydroxy-1-naphthaldehyde. Mononuclear complexes with a metal ligand ratio of 1 : 2 have been prepared with Co(II), Ni(II), Cu(II) and Zn(II) salts. Their structures have been elucidated on the basis of elemental analyses, IR, ^{1}H- and ^{13}C-NMR spectra, UV-Vis spectra, magnetic susceptibility measurements, molar conductivity and thermogravimetric analyses (TGA).

Keywords: Schiff bases; Transition metal complexes

1. Introduction

Schiff-base transition metal complexes are of interest in inorganic chemistry and have been studied extensively [1]. Complexes of transition metal ions with bidentate Schiff bases containing nitrogen and oxygen donor atoms play an important role in biological systems and represent interesting models for metalloenzymes that catalyze the reduction of nitrogen and oxygen [2]. Schiff-base metal complexes have industrial, antifungal and biological applications [3]. Research on liquid crystals discovered that introduction of lateral polar hydroxy groups enhances the molecular polarizability as well as stabilizing the liquid crystalline compounds [4]. Although the redox behavior of a number of metal complexes containing Schiff-base ligands is known, the electrochemical properties of such complexes are not completely clear [5]. Also Schiff bases and their complexes in aqueous solutions have interesting and important properties [6]. In this article,

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Journal of Coordination Chemistry
ISSN 0095-8972 print: ISSN 1029-0389 online © 2005 Taylor & Francis Group Ltd
DOI: 10.1080/00958970500122565

we report the synthesis and characterization of a new Schiff-base ligand and its complexes with cobalt(II), nickel(II), copper(II) and zinc(II).

2. Experimental

Elemental analyses (C, H, N) were carried out using LECO-932 CHNSO by the Technical and Scientific Research Council of Turkey, TUBITAK. IR spectra were recorded on a Mattson 1000 FT-IR Spectrometer as KBr pellets. ^{1}H- and ^{13}C-NMR spectra were recorded on a Bruker GmbH Dpx-400 MHz High Performance Digital FT-NMR Spectrometers. Electronic spectra were obtained on a Shimadzu 1240 UV Spectrometer at Hacettepe University. Magnetic susceptibilities were determined on a Sherwood Scientific Magnetic Susceptibility Balance (Model MK1) at room temperature using $Hg[Co(SCN)_4]$ as a calibrant; diamagnetic corrections were calculated from Pascal's constants. The melting points were determined on a Gallenkamp melting point apparatus. Molar conductances of the Schiff base and its transition metal complexes were determined at room temperature using a CMD 750 WPA conductivity meter. TGA curves were recorded on a Shimadzu TG-50 thermo balance.

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2.1. *Naphthyliden-p-aminoacetophenoneoxime (LH)*

To a solution of (1.502 g, 10 mmol) *p*-aminoacetophenoneoxime [7] in 15 mL absolute ethanol (1.722 g, 10 mmol) of 2-hydroxy-1-naphthaldehyde and (0.01 mg) *p*-toluene sulfonic acid dissolved in 15 mL absolute ethanol were added dropwise at 60°C with continuous stirring and monitoring of the course of the reaction with IR. The precipitate was filtered, washed with cold ethanol and diethylether several times and crystallized from acetone–water and dried at 60°C to a constant weight.

[LH] was obtained in 79% (2.40 g) yield. IR (cm^{-1}): 3300–3430 (oxime O–H), 3198–3250 (phenolic O–H), 1621 (phenolic C=N), 1598 (oxime C=N), 1310 (C–O), 1011 (N–O). ^{1}H NMR ($CDCl_3$-DMSO-d_6, δ): 13.54 (s, 1H, H^a), 10.74 (s, 1H, H^b), 8.64 (s, 1H, H_9), 7.66–6.83 (m, 10H, Arom-H), 2.17 (s, 3H, H_1). ^{13}C NMR ($CDCl_3$-DMSO-d_6, δ): 162.74 (C_{19}), 160.20 (C_9), 152.83 (C_6), 147.93 (C_2), 138.24 (C_{17}), 137.82 (C_3), 136.14 (C_{13}), 133.11 (C_{11}), 132.74 (C_4 and C_8), 128.40 (C_{15}), 127.33 (C_{12}), 126.51 (C_{16}), 123.80 (C_5 and C_7), 122.61 (C_{18}), 119.88 (C_{10}), 117.74 (C_{16}), 11.34 (C_1). λ_{max} [ε, $L\,mol^{-1}\,cm^{-1}$]: 390 [2.30×10^3] (azomethine $n \rightarrow \pi^*$).

2.2. *Bis(p-aminoacetophenoneoxime-naphthylaldiminato)cobalt(II) Co(L)$_2$*

A mixture of $Co(AcO)_2 \cdot 4H_2O$ (0.125 g, 0.50 mmol) and LH (0.304 g, 1.00 mmol) was heated to 50°C for 15 h under reduced pressure in a sealed tube containing 40 cm^3 absolute ethanol. The complex was obtained immediately upon addition of the metal salt solution. The reaction mixture was cooled and the solvent removed on a vacuum line. The residue was washed several times with water, cold ethanol and cold diethyl ether. The complex was left to dry *in vacuo* for several hours.

[$Co(L)_2$] was obtained in 71% (0.24 g) yield. IR (cm^{-1}): 3300–3430 (oxime O–H), 1606 (phenolic C=N), 1595 (oxime C=N), 1318 (C–O), 1007 (N–O). λ_{max} [ε, $L\,mol^{-1}\,cm^{-1}$]: 381 [3.50×10^3] (azomethine $n \rightarrow \pi^*$), 451 [2.00×10^3] (C–T), 686 [191] (d–d).

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2.3. *Bis(p-aminoacetophenoneoxime-naphthylaldiminato)nickel(II) Ni(L)$_2$*

A similar procedure as for Co(L)$_2$ was employed but the reaction period was 10 h.

[Ni(L)$_2$] was obtained in 64% (0.21 g) yield. IR (cm^{-1}): 3300–3430 (oxime O–H), 1613 (phenolic C=N), 1595 (oxime C=N), 1322 (C–O), 1007 (N–O). λ_{max} [ε, L mol^{-1} cm^{-1}]: 380 [3.17×10^3] (azomethine $n \rightarrow \pi^*$), 443 [2.90×10^3] (C–T), 645 [173] (d–d).

2.4. *Bis(p-aminoacetophenoneoxime-naphthylaldiminato)copper(II) Cu(L)$_2$*

A similar procedure as for Co(L)$_2$ was employed but the reaction period was 8 h.

[Cu(L)$_2$] was obtained in 76% (0.26 g) yield. IR (cm^{-1}): 3300–3430 (oxime O–H), 1605 (phenolic C=N), 1598 (oxime C=N), 1314 (C–O), 1007 (N–O). λ_{max} [ε, L mol^{-1} cm^{-1}]: 378 [4.00×10^3] (azomethine $n \rightarrow \pi^*$), 440 [1.80×10^3] (C–T), 580 [138] (d–d).

2.5. *Bis(p-aminoacetophenoneoxime-naphthylaldiminato)zinc(II) Zn(L)$_2$*

A similar procedure as for Co(L)$_2$ was employed but the reaction period was 20 h.

[Zn(L)$_2$] was obtained in 70% (0.24 g) yield. IR (cm^{-1}): 3300–3430 (oxime O–H), 1600 (phenolic C=N), 1595 (oxime C=N), 1316 (C–O), 1007 (N–O). ^{1}H NMR (CDCl$_3$-DMSO-d$_6$, δ): 10.75 (s, 2H, H^b), 8.58 (s, 2H, H$_9$), 7.65–6.77 (m, 20H, Arom-H), 2.23 (s, 6H, H$_1$). ^{13}C NMR (CDCl$_3$-DMSO-d$_6$, δ): 165.81 (C$_{19}$), 162.90 (C$_9$), 154.10 (C$_6$), 147.98 (C$_2$), 138.00 (C$_{17}$), 137.71 (C$_3$), 136.55 (C$_{13}$), 133.11 (C$_{11}$), 132.94 (C$_4$ and C$_8$), 133.01 (C$_{17}$), 128.64 (C$_{15}$), 127.33 (C$_{12}$), 126.51 (C$_{14}$), 123.80 (C$_5$ and C$_7$), 122.61 (C$_{18}$), 119.88 (C$_{10}$), 117.74 (C$_{16}$), 11.28 (C$_1$). λ_{max} [ε, L mol^{-1} cm^{-1}]: 376 [2.60×10^3] (azomethine $n \rightarrow \pi^*$), 462 [3.05×10^3] (C–T).

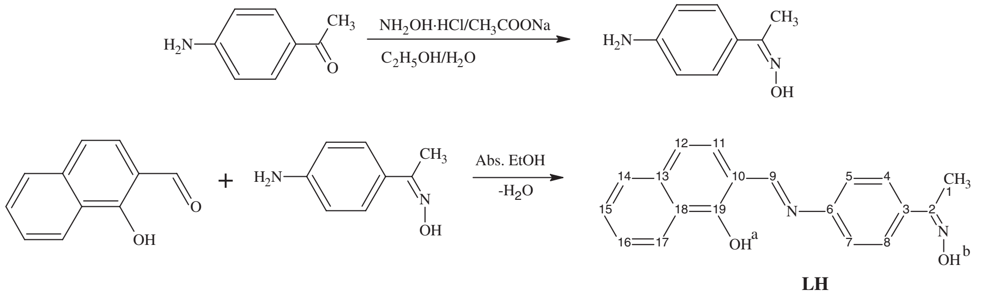
3. Results and discussion

Microanalytical, molar conductance and magnetic susceptibility data of the ligand and its complexes are given in table 1.

Table 1. Analytical and physical data of the ligand and the complexes.

Compounds	Formula (F.W) g mol^{-1}	Color	m.p. (°C)	Yield (%)	μ_{eff} (B.M.)	Λ_M (Ω^{-1} cm^2 mol^{-1})	Elemental analysis calculated (found) (%)		
							C	H	N
LH	$C_{19}H_{16}N_2O_2$ (304.34)	Dark orange	244	79	–	2.6	74.98 (75.35)	5.30 (5.66)	9.20 (8.81)
Co(L)$_2$	$CoC_{38}H_{30}N_4O_4$ (665.60)	Dark red	> 300	71	3.96	4.1	68.57 (68.23)	4.54 (4.90)	8.42 (8.73)
Ni(L)$_2$	$NiC_{38}H_{30}N_4O_4$ (665.36)	Light green	257[a]	64	3.10	3.2	68.60 (68.94)	4.54 (4.36)	8.42 (8.09)
Cu(L)$_2$	$CuC_{38}H_{30}N_4O_4$ (670.22)	Brown	241	76	2.05	4.8	68.10 (67.75)	4.51 (4.82)	8.36 (7.99)
Zn(L)$_2$	$ZnC_{38}H_{30}N_4O_4$ (672.06)	Orange	258	70	dia	5.5	67.91 (68.28)	4.50 (4.90)	8.34 (7.95)

[a] Dec.



Scheme 1. Structure of the ligand.

The reaction step for the synthesis of naphthyliden-*p*-aminoacetophenoneoxime (LH) is given in scheme 1. The first step is synthesis of *p*-aminoacetophenoneoxime from the reaction of *p*-aminoacetophenone in hydroxylamine hydrochloride and sodium acetate. In the second step, the LH ligand was synthesized by condensation of *p*-aminoacetophenoneoxime with 2-hydroxy-1-naphthaldehyde (scheme 1). The structure of the ligand and its complexes were determined by elemental analysis, IR techniques, ^{1}H- and ^{13}C-NMR spectroscopy, UV-Vis spectra, magnetic susceptibility measurements, conductivity measurements and thermogravimetric analyses.

Reactions of the transition metal salts with the Schiff base are indicated by the following equations.

$$2\mathrm{LH} + \mathrm{Co(CH_3COO)_2 \cdot 4H_2O} \rightarrow \mathrm{Co(L)_2} + 2\mathrm{CH_3COOH} + 4\mathrm{H_2O}$$
$$2\mathrm{LH} + \mathrm{Ni(CH_3COO)_2 \cdot 4H_2O} \rightarrow \mathrm{Ni(L)_2} + 2\mathrm{CH_3COOH} + 4\mathrm{H_2O}$$
$$2\mathrm{LH} + \mathrm{Cu(CH_3COO)_2 \cdot H_2O} \rightarrow \mathrm{Cu(L)_2} + 2\mathrm{CH_3COOH} + \mathrm{H_2O}$$
$$2\mathrm{LH} + \mathrm{Zn(CH_3COO)_2 \cdot 2H_2O} \rightarrow \mathrm{Zn(L)_2} + 2\mathrm{CH_3COOH} + 2\mathrm{H_2O}$$

All complexes are soluble in DMSO and DMF. The metal-to-ligand ratio was found to be 1 : 2 by elemental analyses. The probable structure of the complexes is shown in figure 1.

3.1. *IR spectra*

The tentative assignment of the important IR bands of the Schiff base under investigation and its corresponding metal complexes are recorded in the experimental section. The ligand contains four potential donor sites: (1) the phenolic oxygen, (2) the azomethine nitrogen, (3) the oxime oxygen, (4) the oxime nitrogen. The broad band that appeared in the IR spectrum of LH at 3198–3250 $\mathrm{cm^{-1}}$ is characteristic for the stretching vibration of intermolecular hydrogen bonded –OH [8, 9]. The band at 1310 $\mathrm{cm^{-1}}$ in the IR spectrum of the synthesized ligand is ascribed to the phenolic C–O stretching vibration [10]. The band is shifted to higher frequency by 4–12 $\mathrm{cm^{-1}}$ due to O-metal coordination [11]. A strong band observed in the IR spectrum of the free ligand at 1621 $\mathrm{cm^{-1}}$ is attributed to the C=N stretch. This band shifts to ca 1600–1613 $\mathrm{cm^{-1}}$ in the spectra of all the complexes, indicating coordination of the azomethine nitrogen

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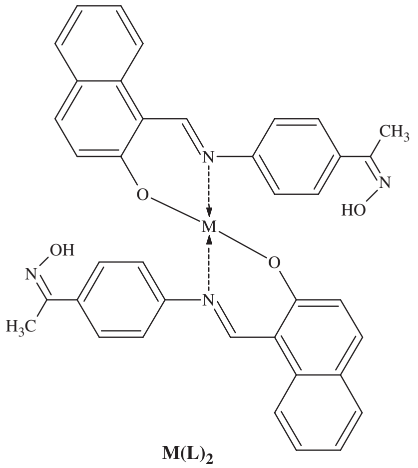

M = Co(II), Ni(II), Cu(II) and Zn(II)

Figure 1. Suggested structure of the tetrahedral complexes.

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atom to the metals [12]. The practically unchanged O–H around 3300–3430 [13–15] and C=N at 1598 cm^{-1} [16–18] indicates that the oxime group itself does not coordinate [19].

3.2. ^{1}H- and ^{13}C-NMR spectra

The ^{1}H- and ^{13}C-NMR spectral results obtained for LH and the zinc(II) complex in $CDCl_3$/DMSO-d_6, together with the assignments, are given in the section on synthesis of the compounds. ^{1}H-NMR spectra of the ligand obtained in $CDCl_3$/DMSO-d_6, shows a singlet at 13.54 ppm, which disappears upon addition of D_2O and is assigned to phenolic group (–OH) [20]. In the ^{1}H-NMR spectra of LH the resonance observed at 8.64 ppm is assigned to the azomethine proton (CH=N) [21]. In the ^{13}C-NMR spectra, the C–OH, CH=N and C–N groups in the ligand are at 162.74, 160.20 and 152.83 ppm, respectively. The ^{1}H-NMR spectrum of zinc(II) complex shows no OH peaks as expected indicating the coordination by phenolic oxygen to the metal ion after deprotonation. Coordination of the azomethine nitrogen is inferred by the upfield shift of the (CH=N) proton signal from 8.64 ppm in the ligand to 8.58 ppm in the complex [22]. ^{13}C-NMR spectrum of zinc(II) complex have C–OH, CH=N and C–N carbon atoms at 165.81, 162.90 and 154.10 ppm, respectively. The rest of the carbon atoms are similar to the free ligand. The signals corresponding to the =N–OH proton and C=N–OH carbon (both in oxime) [23–25] groups are unchanged in the ^{1}H- and ^{13}C-NMR spectra of the complexes indicating that these oxime groups do not take part in complexation.

3.3. UV-Vis spectra

The electronic spectra of all complexes were recorded in 10^{-3} M DMF at room temperature. The Schiff base is characterized mainly by two absorption bands in the region 275–400 nm. In the spectra of the Schiff-base ligand, the band at 300 nm is attributed to a benzene $\pi \rightarrow \pi^*$ transition. The band at 390 nm is assigned to the

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imino $\pi \rightarrow \pi^*$ transition. The longer wavelength band is assigned to intermolecular charge transfer while the shorter wavelength is due to $\pi \rightarrow \pi^*$ within the C=N band influenced by C–T interaction [26]. The bands in the electronic spectra of Co(II) complex at 451 nm ($\varepsilon = 2.00 \times 10^{-3}$) and 686 nm ($\varepsilon = 191$) can be assigned to the C–T and $^4A_2 \rightarrow {}^4T_1$ transitions respectively, in the tetrahedral ligand field [27]. The electronic spectra of the Ni(II) complex show two absorption bands, at 443 nm ($\varepsilon = 2.90 \times 10^{-3}$) and 645 nm ($\varepsilon = 173$), due to the C–T and $^3A_2 \rightarrow {}^3T_2$ transitions respectively, observed for tetrahedral Ni(II) complex [28]. The spectra of the Cu(II) have a less intense shoulder at 580 nm ($\varepsilon = 138$), which is assigned as a d–d transition of the metal ion, probably due to the $^2T_2 \rightarrow {}^2E(G)$ transition of tetrahedral geometry [29]. The electronic spectrum of the Zn(II) complex shows an absorption band at 462 nm ($\varepsilon = 3.05 \times 10^{-3}$) attributed to the C–T transition, which is compatible with this complex having a tetrahedral structure [30].

3.4. *Magnetic properties*

The magnetic moments of the complexes were measured at room temperature. The Co(II), Ni(II) and Cu(II) complexes are paramagnetic and their magnetic moments are 3.96, 3.10 and 2.05 B.M., respectively [31, 32]. The Zn(II) complex is diamagnetic.

3.5. *Conductivity*

The complexes are non-electrolytes as shown by their molar conductivity (Λ_M) measurements in 10^{-4} M DMSO solutions, which are in the range 3.2–5.5 Ω^{-1} cm^2 mol^{-1} [33].

3.6. *Thermal studies*

The thermogravimetric (TGA), curves for the complexes were obtained at a heating rate of 10°C min^{-1} and a 30 mL min^{-1} flowing nitrogen atmosphere over a temperature range of 20–800°C. TGA data of the ligand and its metal complexes are given in table 2. The decomposition temperature and the weight losses of the complexes were calculated from TGA data. The ligand is stable up to 176°C where decomposition starts and is completed at 595°C. All complexes and the ligand decompose

Table 2. TGA data of the ligand and the complexes.

	Decomposition temperature (%) Calculated (found)			
Compounds	First step	Second step	Weight loss (%) Calculated (found)	Residue (%) Calculated (found)
LH	176–275	275–595		
	51.32 (50.09)	48.68 (47.70)	(97.79)	(2.21)
Co(L)$_2$	242–286	286–451		
	19.82 (18.40)	68.92 (70.18)	88.74 (88.58)	11.26 (11.42)
Ni(L)$_2$	242–297	297–402		
	19.84 (18.79)	68.93 (70.93)	88.77 (89.72)	11.23 (10.28)
Cu(L)$_2$	225–250	250–483		
	11.64 (10.08)	76.49 (78.97)	88.13 (89.05)	11.87 (10.95)
Zn(L)$_2$	210–294	294–518		
	19.64 (20.44)	68.25 (67.53)	87.89 (87.97)	12.11 (12.03)

in two steps (table 2) at different temperature ranges [34, 35]. In addition, the thermal stability of all complexes increases in the order: Cu < Ni < Co < Zn. When the complexes are heated to higher temperatures, they decompose to give MO oxides [36].

Acknowledgements

The support of the Management Unit of Scientific Research Projects of Firat University (FUBAP) under research project No: 835 is gratefully acknowledged.

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