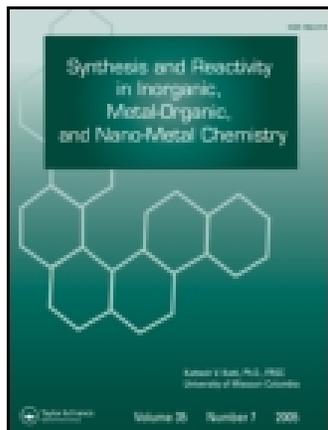


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# Synthesis and Characterization of New Schiff Bases and Their Cobalt(II), Nickel(II), Copper(II), Zinc(II), Cadmium(II) and Mercury(II) Complexes

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Nickel(II), copper(II), cobalt(II), zinc(II), cadmium(II) and mercury(II) complexes chelates containing the Schiff base ligands methyliminoisonitroso-2-acetylnaphthalene (HL<sup>1</sup>) and benzyliminoisonitroso-2-acetylnaphthalene (HL<sup>2</sup>) have been prepared. The results suggest that the Schiff bases function as bidentate ligands and indicate a mononuclear structure for the Cu(II) complexes with square-planar geometry. The Ni(II), Zn(II), Hg(II) and Cd(II) complexes are of tetrahedral geometry and the Co(II) complexes with octahedral geometry with water molecules at the axial positions. These compounds have been characterized by elemental analyses, magnetic susceptibility, mass, IR, UV, <sup>1</sup>H and <sup>13</sup>C NMR spectroscopy.

**Keywords** Schiff base ligands, spectra, transition metal complexes

## INTRODUCTION

The formation of Schiff bases by the condensation of amines with aldehydes or ketones is well known (Amirnasr et al., 2002; Deveci and İrez, 1995; Dreos et al., 2003; Garg and Kumar, 2003; Karataş and Uçan, 1998; Köksal et al., 1996; Mercimek et al., 1999; Mokles and Elzahr, 2001). Ligands derived by condensation of a primary amine and an active carbonyl group contain the azomethine group and form stable complexes with metal ions, especially if the amine and the carbonyl compound contain a second functional group sufficiently near the site of condensation to form a five- or six-membered chelate ring upon chelation (Park et al., 1998; Ray et al., 2003; Tarafder et al., 2002; Tümer et al., 1997; Tuna et al., 2003). In addition, a great many Schiff base complexes with

metal ions have also provoked wide interest because they possess a diverse spectrum of biological and pharmaceutical activities, such as antitumor and antioxidative activities, as well as the inhibition of lipid peroxidation, etc (Uçan et al., 1998; Yang et al., 2000; Yıldırım et al., 2003).

The present investigation is concerned with the preparation and characterization of Ni(II), Cu(II), Co(II), Zn(II), Cd(II) and Hg(II) chelates with the bidentate Schiff bases derived from amines with isonitroso-2-acetylnaphthalene (Figure 1).

## EXPERIMENTAL

### Materials and Measurements

Isonitroso-2-acetylnaphthalene was prepared according to published procedures (Tümer et al., 1997; Tuna et al., 2003). The amine compounds (methylamine and benzylamine) were obtained from Fluka and were used without further purification. All solvents were obtained from E. Merck or Fluka. The metal salts were purchased from E. Merck and were used as received. Elemental analyses (C, H, N) were performed using a Carlo-Erba 1106 elemental analyzer. UV-Vis. spectra were recorded on a Shimadzu UV-160A spectrophotometer. The samples were dissolved in DMF and the spectra were recorded in the 800–200 nm range. The magnetic moments of the complexes were measured by the Gouy method on a type D-104 Newport Instrument. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded on a Varian XL 200 spectrometer with the samples dissolved in DMSO-d<sub>6</sub> using TMS as internal standard. The IR spectra were recorded in KBr pellets in the range 4000–400 cm<sup>-1</sup> on a Pye-Unicam SP 1025 model FT-IR spectrometer.

### Synthesis of Schiff Base Ligand

Methyliminoisonitroso-2-acetylnaphthalene [HL<sup>1</sup>] was prepared by a condensation method. Methylamine (0.18 g, 6 mmol) was dissolved in MeOH (10 mL) and was added to a

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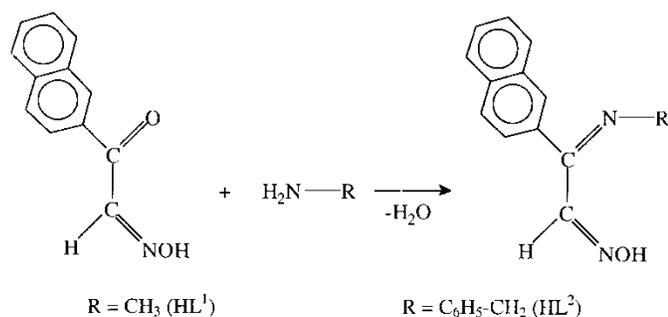


FIG. 1. Structures of the ligands.

solution of isonitroso-2-acetylnaphthalene (1.19 g, 6 mmol) in 20 mL of MeOH. The reaction mixture was stirred for 2 h and then left overnight at room temperature. The solid reaction product was filtered and washed with cold ethanol. The ligand was recrystallized from dichloromethane/ethanol (1:1) and

dried in a vacuum desiccator over  $\text{CaCl}_2$ . Benzyliminoisonitroso-2-acetylnaphthalene was prepared in a similar fashion.

### Synthesis of Complexes

The complexes were synthesized by a modification of literature preparations (Deveci and İrez, 1995; Mokles and Elzaher, 2001; Tarafder et al., 2002; Tümer et al., 1997). A solution of  $\text{Ni}(\text{AcO})_2 \cdot 4\text{H}_2\text{O}$  (0.124 g, 0.5 mmol),  $\text{Cu}(\text{AcO})_2 \cdot \text{H}_2\text{O}$  (0.099 g, 0.5 mmol),  $\text{Co}(\text{AcO})_2 \cdot 4\text{H}_2\text{O}$  (0.124 g, 0.5 mmol),  $\text{Zn}(\text{AcO})_2 \cdot 2\text{H}_2\text{O}$  (0.109 g, 0.5 mmol),  $\text{Cd}(\text{AcO})_2 \cdot 2\text{H}_2\text{O}$  (0.133 g, 0.5 mmol) or  $\text{Hg}(\text{AcO})_2$  (0.159 g, 0.5 mmol), in EtOH (10 mL) was added to a solution of  $\text{HL}^1$  (0.212 g, 1 mmol) in EtOH. The mixture was refluxed for 3 h at  $70^\circ\text{C}$ . The precipitated complex was filtered, washed with cold EtOH and dried in a vacuum desiccator over  $\text{CaCl}_2$ . The same experimental procedure was followed in the preparation of Ni(II), Cu(II), Co(II), Zn(II), Cd(II) and Hg(II) Complexes of  $[\text{HL}^2]$ .

TABLE 1  
Physical properties and elemental analysis of the ligands and complexes

Compound	FW.	Color	Yield (%)	M.p. ( $^\circ\text{C}$ )	$\mu_{\text{eff}}$ (B.M.)	Analysis found (calcd.) %		
						C	H	N
$\text{HL}^1$ ( $\text{C}_{13}\text{H}_{12}\text{N}_2\text{O}$ )	212	Yellow	90	84		73.56 (73.39)	5.69 (5.77)	13.19 (13.35)
$[\text{Ni}(\text{L}^1)_2] \text{C}_{26}\text{H}_{22}\text{NiN}_4\text{O}_2$	481	Green	92	251	3.5	64.9 (64.74)	4.6 (4.43)	11.64 (11.79)
$[\text{Cu}(\text{L}^1)_2] \text{C}_{26}\text{H}_{22}\text{CuN}_4\text{O}_2$	486	Brown	81	283	1.66	64.25 (64.15)	4.56 (4.46)	11.52 (11.26)
$[\text{Co}(\text{L}^1)_2(\text{H}_2\text{O})_2] \text{C}_{26}\text{H}_{26}\text{CoN}_4\text{O}_4$	517	Red	83	220	4.7	60.35 (60.28)	5.06 (4.92)	10.82 (10.5)
$[\text{Zn}(\text{L}^1)_2] \text{C}_{26}\text{H}_{22}\text{ZnN}_4\text{O}_2$	487	Light yellow	95	137	Diamag.	64.01 (63.91)	4.54 (4.48)	11.48 (11.22)
$[\text{Cd}(\text{L}^1)_2] \text{C}_{26}\text{H}_{22}\text{CdN}_4\text{O}_2$	534	Yellow	79	196	Diamag.	58.38 (58.3)	4.14 (4.04)	10.47 (10.35)
$[\text{Hg}(\text{L}^1)_2] \text{C}_{26}\text{H}_{22}\text{HgN}_4\text{O}_2$	623	Orange	80	126	Diamag.	50.12 (49.97)	3.55 (3.32)	8.99 (8.87)
$\text{HL}^2$ $\text{C}_{19}\text{H}_{16}\text{N}_2\text{O}$	288	Yellow	86	79		79.14 (78.99)	5.29 (5.23)	9.71 (9.6)
$[\text{Ni}(\text{L}^2)_2] \text{C}_{38}\text{H}_{30}\text{NiN}_4\text{O}_2$	633	Green	82	284	4.0	72.06 (71.88)	4.77 (4.61)	8.84 (8.78)
$[\text{Cu}(\text{L}^2)_2] \text{C}_{38}\text{H}_{30}\text{CuN}_4\text{O}_2$	638	Brown green	76	197	1.75	71.51 (71.38)	4.73 (4.68)	8.77 (8.67)
$[\text{Co}(\text{L}^2)_2(\text{H}_2\text{O})_2] \text{C}_{38}\text{H}_{34}\text{CoN}_4\text{O}_4$	669	Red	80	184	5.7	68.15 (67.97)	5.11 (5.03)	8.36 (8.24)
$[\text{Zn}(\text{L}^2)_2] \text{C}_{38}\text{H}_{30}\text{ZnN}_4\text{O}_2$	640	Light yellow	92	141	Diamag.	71.34 (71.26)	4.72 (4.64)	8.75 (8.68)
$\text{Cd}(\text{L}^2)_2 \text{C}_{38}\text{H}_{30}\text{CdN}_4\text{O}_2$	687	Yellow	89	163	Diamag.	66.42 (66.39)	4.4 (4.26)	8.15 (8.09)
$[\text{Hg}(\text{L}^2)_2] \text{C}_{38}\text{H}_{30}\text{HgN}_4\text{O}_2$	775	Orange	86	139	Diamag.	58.87 (58.7)	3.9 (3.75)	7.22 (7.14)

TABLE 2  
 Characteristic IR bands<sup>a</sup> of the ligands and their complexes (KBr, cm<sup>-1</sup>)

Compounds	IR spectra (cm <sup>-1</sup> )							Electronic data in DMSO	Molecular ion
	$\nu(\text{O-H})$	$\text{N}(\text{C-H})^a$	$\nu(\text{C-H})$	$\nu(\text{C}=\text{N})$	$\nu(\text{N-O})$	$\text{N}(\text{M-N})$	$\text{N}(\text{M-O})$	$\lambda_{\text{max}}(\text{cm}^{-1})$	found (calc.) <sup>a</sup>
HL <sup>1</sup>	3230 b	3100 w	2770 w	1645–1625 s	990 w				212.1 (212)
[Ni(L <sup>1</sup> ) <sub>2</sub> ]		3055 w	2820 w	1614–1569 s	925 w	535 w	482 w	15.625	
[Cu(L <sup>1</sup> ) <sub>2</sub> ]		3050 w	2800 w	1622–1599 s	924 w	540 w	475 w	15.350	
[Co(L <sup>1</sup> ) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]	3420 b	3070 w	2750 w	1629–1605 s	920 w	481 w	436 w	16.452	517.1 (517)
[Zn(L <sup>1</sup> ) <sub>2</sub> ]		3050 w	2700 w	1613–1576 s	913 m	580 w	481 w	29.186	
[Cd(L <sup>1</sup> ) <sub>2</sub> ]		3060 w	2930 w	1617–1579 s	924 w	485 w	431 w		
[Hg(L <sup>1</sup> ) <sub>2</sub> ]		3045 w	2900 w	1621–1585 s	925 m	489 w	437 w		
HL <sup>2</sup>	3208 b	3050 w	2700 w	1645–1621 s	990 w				288.1(288)
[Ni(L <sup>2</sup> ) <sub>2</sub> ]		3055 w	2850 w	1605–1585 s	925 w	490 w	430 w		633.3 (633)
[Cu(L <sup>2</sup> ) <sub>2</sub> ]		3050 w	2820 w	1600–1580 s	930 w	500 w	400 w		
[Co(L <sup>2</sup> ) <sub>2</sub> (H <sub>2</sub> O) <sub>2</sub> ]	3415 b	3075 w	2880 w	1621–1600 s	925 w	484 w	431 w		
[Zn(L <sup>2</sup> ) <sub>2</sub> ]		3085 w	2860 w	1620–1576 s	916 w	481 w	420 w		
Cd(L <sup>2</sup> ) <sub>2</sub>		3070 w	2830 w	1622–1585 s	940 m	530 w	425 w		
[Hg(L <sup>2</sup> ) <sub>2</sub> ]		3046 w	2825 w	1614–1585 s	916 m	481 w	420 w		

<sup>a</sup>b: broad, s: strong, m: medium, w: weak.

TABLE 3  
NMR spectral data (in ppm) of the ligands and their complexes

Compounds	<sup>1</sup> H NMR signals			Aromatic			<sup>13</sup> C NMR signals		
	C=NOH	CH=N	CH <sub>3</sub>	CH <sub>2</sub>	proton	C=NO	CH=N	CH <sub>3</sub>	CH <sub>2</sub>
HL <sup>1</sup>	8.90	8.52	2.48		8.38–7.80	189.59	148.58	28.30	
[Ni(L <sup>1</sup> ) <sub>2</sub> ]		8.46	2.38		8.15–7.36	186.70	147.25	20.12	
HL <sup>2</sup>	8.62	8.14		3.85	8.02–7.18	188.57	147.54		44.43
[Ni(L <sup>2</sup> ) <sub>2</sub> ]		8.08		3.16	7.96–7.06	185.12	142.34		40.00

## RESULTS AND DISCUSSION

The reaction of isonitroso-2-acetylnaphthalene with methylamine or benzylamine in MeOH gave the desired substituted methyliminoisonitroso-2-acetylnaphthalene (HL<sup>1</sup>) and benzyliminoisonitroso-2-acetylnaphthalene (HL<sup>2</sup>) in a single step (Figure 1). The ligands are soluble in EtOH, MeOH, chloroform and diethyl ether. The addition of Ni(II), Cu(II), Co(II), Zn(II), Hg(II) or Cd(II) acetate dissolved in methanol to an alcoholic solution of the ligands gave colored complexes. All of the complexes are soluble in DMSO, THF and DMF, but insoluble in the other common organic solvents. The compositions of the Schiff bases and their complexes have been verified by elemental analyses (Table 1).

In the IR spectral data of the ligands, bands at 3230–3208, 3100–3050 cm<sup>-1</sup>, 1645–1621 cm<sup>-1</sup> and 990–980 cm<sup>-1</sup> belong to O–H, C–H<sub>(arom)</sub>, C=N and N–O stretching bands, respectively. Evidence of nitrogen bonding of the azomethine C=N group to the metal ion stems from the shift of the  $\nu(\text{C}=\text{N})$  frequency to lower frequencies by 16–41 cm<sup>-1</sup> in all of the complexes (Amirnasr et al., 2002; Deveci and İrez, 1995; Dreos et al., 2003; Garg and Kumar, 2003; Mokles and Elzaher, 2001; Tarafder et al., 2002; Tümer et al., 1997). New bands in the complexes at 580–420 cm<sup>-1</sup> are attributed to the  $\nu(\text{M}-\text{N})$  and  $\nu(\text{M}-\text{O})$  vibrations, respectively (Mokles and Elzaher, 2001; Tarafder et al., 2002; Uçan et al., 1998; Zishen et al., 1990).

The <sup>1</sup>H NMR spectra of the ligands exhibited a singlet peak for the OH protons of the oxime groups at 8.90–8.62 ppm. For the N=CH protons, a singlet occurs in the 8.52–8.14 ppm region (Karataş and Uçan, 1998; Mercimek et al., 1999; Tümer et al., 1997; Tuna et al., 2003). The broad multiplet at 8.38–7.18 ppm, has been assigned to the aromatic ring protons. A singlet observed at 2.48 ppm corresponds to the methyl protons. The methylene protons at 3.85 ppm (Deveci and İrez, 1995; Köksal et al., 1996; Mercimek et al., 1999; Tuna et al., 2003).

In the <sup>13</sup>C NMR spectra of the ligands, the signals at 189.59–188.57 and 148.58–147.54 ppm are attributed to the carbon atoms of the oxime groups and the carbon atoms of the azomethine groups, respectively (Park et al., 1998; Ray et al., 2003; Tarafder et al., 2002). All the signals in the

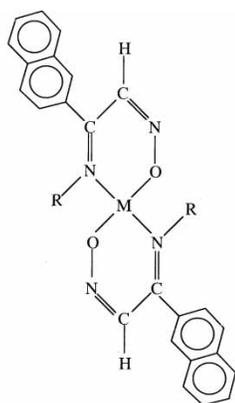
135.88–125.75 ppm range are assigned to the carbon atoms of the aromatic rings. The signals observed at 28.30 and 44.43 ppm may be attributed to the carbon atoms of the CH<sub>3</sub> and CH<sub>2</sub> groups, respectively (Köksal et al., 1996; Tarafder et al., 2002; Zishen et al., 1990).

The mass spectra of the ligand HL<sup>1</sup> gave molecular ion at m/z 212 and it's the Co(II) complex at m/z 517. Molecular ion of HL<sup>2</sup> appeared at m/z 288 and its Ni(II) complex showed peak at m/z 633.

The electronic spectra of all the complexes were recorded in 10<sup>-3</sup> M DMF solution at room temperature. The free Schiff base exhibits two absorption bands in the 252–275 and 305–324 nm regions. These bands are attributed to  $\pi \rightarrow \pi^*$  transitions, the first being due to the aromatic ring and the second the imino group. In the complexes, the imino  $\pi \rightarrow \pi^*$  transition is shifted to a longer wave length as a consequence of coordination to the metal, confirming the formation of Schiff base metal complexes.

The electronic spectra of the Cu(II) complex shows an absorption band at 15.350 cm<sup>-1</sup> attributed to the <sup>2</sup>B<sub>1g</sub> → <sup>2</sup>A<sub>1g</sub> transition, which is compatible with this complex having a square-planar structure. The electronic spectra of the Ni(II) complexes exhibit two bands at 15.625 cm<sup>-1</sup> and 34.483 cm<sup>-1</sup>. The former is probably due to the <sup>3</sup>T<sub>1</sub> → <sup>3</sup>T<sub>1</sub>(P) transition and the latter due to the charge transfer transition of tetrahedral geometry. The lowest energy band, <sup>3</sup>T<sub>1</sub> → <sup>3</sup>T<sub>2</sub>, is not observed in the spectrum. The Co(II) complex show two bands at 16.452 cm<sup>-1</sup> and 18.530 cm<sup>-1</sup> corresponding to the transitions <sup>4</sup>T<sub>1</sub>(F) → <sup>4</sup>A<sub>2</sub>(F) and <sup>4</sup>T<sub>1</sub>(F) → <sup>4</sup>T<sub>1</sub>(P), respectively. All these findings favor an octahedral geometry for this complex. The spectrum of the Zn(II) complex shows an absorption band at 29.186 cm<sup>-1</sup> attributed to the L → M (charge transfer) transition, which is compatible with this complex having a tetrahedral structure.

The magnetic moment data of the complexes are given in Table 1. The values 1.66 and 1.75 B.M. observed for [Cu(L<sup>1</sup>)<sub>2</sub>] and [Cu(L<sup>2</sup>)<sub>2</sub>], respectively, reveal the square-planar nature around the Cu(II) ion (Karataş and Uçan, 1998; Köksal et al., 1996; Mercimek et al., 1999). The values of nickel complexes [Ni(L<sup>1</sup>)<sub>2</sub>] and [Ni(L<sup>2</sup>)<sub>2</sub>] are 3.5 and 4.0 B.M., respectively (Köksal et al., 1996; Mercimek et al.,



M = Ni(II), Cu(II), Zn(II), Cd(II), Hg(II) or Co-2H<sub>2</sub>O

FIG. 2. Suggested structures of the metal complexes.

1999; Mokles and Elzaher, 2001), which reveal the tetrahedral geometry around the nickel ion. The values of 4.7 and 5.7 B.M. are typical of octahedral Co(II). The magnetic moment determinations show that the complexes of Zn(II), Hg(II) and Cd(II) are diamagnetic (Tuna et al., 2003; Zishen et al., 1990) (Figure 2).

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