

## Research Article

# Efficient Synthesis and Characterization of Some Novel Nitro-Schiff Bases and Their Complexes of Nickel(II) and Copper(II)

**Hossein Naeimi and Mohsen Moradian**

*Department of Organic Chemistry, Faculty of Chemistry, University of Kashan, Kashan 87317, Iran*

Correspondence should be addressed to Hossein Naeimi; [naeimi@kashanu.ac.ir](mailto:naeimi@kashanu.ac.ir)

Received 22 February 2012; Revised 16 May 2012; Accepted 21 May 2012

Academic Editor: Serife Tokalioglu

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Synthesis and characterization of some new Schiff base ligands derived from various diamines and nitrosalicylaldehyde and their complexes of Ni(II) and Cu(II) are reported. Several spectral techniques such as UV-Vis, FT-IR, and NMR spectra were used to identify the chemical structures of the reported ligands and their complexes. The ligands are found to be bound to the metal atom through the oxygen atoms of the hydroxyl groups and nitrogen atoms of imine groups, which is also supported by spectroscopic techniques. The results obtained by FT-IR and NMR showed that the Schiff base complexes of transition metal (II) have square-planar geometry.

## 1. Introduction

Schiff bases have played an important role in the development of coordination chemistry as they readily form stable complexes with most of the transition metals. The chemistry of Schiff base ligands species has been gaining considerable interest primarily because of their fascinating structural diversities [1–7].

2-Hydroxy Schiff base ligands and their complexes derived from the reaction of salicylaldehyde derivatives with diamines have been extensively studied in great details for their various crystallographic, structural, and magnetic features [8–14]. Schiff base ligands are able to coordinate many different metals, and to stabilize them in various oxidation states, enabling the use of Schiff base metal complexes for a large variety of useful catalytic transformations.

Schiff bases and their coordination compounds are well known to be biologically important and of interest for their antibacterial, antitumour, and antitubercular activities [15]. Furthermore, it has been used as analytical reagent [16–18], polymer-coating, ink, pigment [19], fluorescent materials [20] and catalytic reagents [21]. However, changes in the electronic, steric, and geometric properties of the ligand alter the orbitals at the metal center and thus affect its properties.

The nitro group is a strong electron withdrawing group and, due to its steric effects, has played an important role in affecting the reactivity and enantioselectivities in synthesis as catalysts [22, 23]. In continuation of our research on the preparation of Schiff base ligands [24–27] and their complexes [28–31], we decided to prepare some new Schiff bases and their complexes, including electron withdrawing substituents.


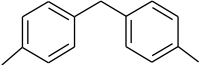
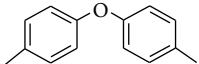
The present paper describes the synthesis and spectroscopic characterization of several nitro-Schiff base ligands and their complexes with transition metal ions under mild conditions. The corresponding materials were characterized by spectroscopic (IR, UV/Vis,  $^1\text{H}$  and  $^{13}\text{C}$  NMR, mass spectra) and physical (melting point) data.

## 2. Experimental

**2.1. Materials.** Chemicals were purchased from the Fluka and Merck Chemical Companies. Solvents were purified by standard methods and dried before use by conventional methods.

**2.2. Apparatus for Analysis and Physical Measurements.** Melting points were taken on a Gallenkamp melting point

TABLE 1: The results of the reaction for preparation of nitro-Schiff bases.

Entry	R (Scheme 1)	Ligand	Time (min)	Yield <sup>a</sup> (%)	M.p (°C)
1	(CH <sub>2</sub> ) <sub>2</sub>	<b>L1</b>	40	91.0	272–274
2	(CH <sub>2</sub> ) <sub>3</sub>	<b>L2</b>	40	90.0	210–213
3	(CH <sub>2</sub> ) <sub>4</sub>	<b>L3</b>	45	88.5	173–176
4	(CH <sub>2</sub> ) <sub>7</sub>	<b>L4</b>	55	88.0	150–152
5	(CH <sub>2</sub> ) <sub>8</sub>	<b>L5</b>	55	85.0	161–164
6		<b>L6</b>	45	92.0	261–264
7		<b>L7</b>	55	92.0	193–195
8		<b>L8</b>	60	94.0	204–206

<sup>a</sup>Isolated yields.

apparatus and are uncorrected. UV-Vis spectra were recorded on Beckman DU-64 spectrophotometers. <sup>1</sup>H NMR spectra were obtained at 400 MHz using a Bruker Avance 400 NMR in CDCl<sub>3</sub> and DMSO-d<sub>6</sub> as the solvents. <sup>13</sup>C NMR (100 MHz) spectra for compounds were obtained on a 400 MHz Avance Bruker spectrometer. The infrared spectra were determined on a Perkin-Elmer 781 spectrophotometer and an Impact 400 Nicolet Magna series FT-IR spectrophotometer.

**2.3. Synthesis of Ligands.** In order to prepare of Schiff base ligands (**L1–L8**), a solution of 5-nitrosalicylaldehyde (4 mmol) in methanol (20 mL) was slowly added over a solution of selected diamine (2 mmol) in the same solvent (20 mL). The mixture was stirred at 30°C; after the reaction is completed, the product precipitated as a yellowish orange solid and the crude solid was filtered off and washed with ethanol twice (2 × 20 mL).

**2.3.1. *N,N'*-Bis(5-Nitrosalicylidene)-1,2-Ethnediamine (L1)** (entry 1, Table 1). 5-Nitrosalicylaldehyde: (0.668 g, 4 mmol); ethanediamine: (0.120 g, 2 mmol); yield: 91%; M.p: 272–274°C. Anal. Calcd.: C, 53.63; H, 3.94; N, 15.64. Found: C, 53.21; H, 3.89; N, 15.55. <sup>1</sup>H NMR, δ(ppm): 14.05–14.50 (s, 2H, O–H), 8.91 (s, 2H, CH=N), 8.58 (d, 2H, Ar–H), 8.17 (dd, 2H, Ar–H), 6.91 (d, 2H, Ar–H), 4.1 (s, 4H, CH<sub>2</sub>). <sup>13</sup>C NMR, δ(ppm): 177.45, 168.73, 136.21, 133.49, 130.62, 123.55, 115.91, 52.28.

**2.3.2. *N,N'*-Bis(5-Nitrosalicylidene)-1,3-Propanediamine (L2)** (entry 2, Table 1). 5-Nitrosalicylaldehyde: (0.668 g, 4 mmol); 1,3-propanediamine: (0.15 g, 2 mmol); yield: 90%; M.p: 210–213°C. Anal. Calcd.: C, 54.84; H, 4.33; N, 15.05. Found: C, 54.39; H, 4.21; N, 14.88. <sup>1</sup>H NMR, δ(ppm): 14.05–14.50 (s, 2H, O–H), 8.91 (s, 2H, HC=N), 8.58 (d, 2H, Ar–H), 8.17 (dd, 2H, Ar–H), 6.91 (d, 2H, Ar–H), 3.91 (t, 4H, CH<sub>2</sub>), 2.38 (q, 2H, CH<sub>2</sub>). <sup>13</sup>C NMR, δ(ppm): 177.45, 168.73, 136.21, 133.49, 130.62, 123.55, 115.91, 52.28, 31.11.

**2.3.3. *N,N'*-Bis(5-Nitrosalicylidene)-1,4-Butanediamine (L3)** (entry 3, Table 1). 5-Nitrosalicylaldehyde: (0.668 g, 4 mmol); 1,4-butanediamine: (0.176 g, 2 mmol); yield: 88%; M.p: 173–176°C. Anal. Calcd.: C, 55.96; H, 4.70; N, 14.50. Found: C, 55.84; H, 4.46; N, 14.81. <sup>1</sup>H NMR, δ(ppm): 14.18–14.78 (s, 2H, O–H), 8.83 (s, 2H, CH=N), 8.50 (d, 2H, Ar–H), 8.21 (dd, 2H, Ar–H), 6.89 (d, 2H, Ar–H), 3.83 (t, 4H, CH<sub>2</sub>), 2.50 (q, 4H, CH<sub>2</sub>). <sup>13</sup>C NMR, δ(ppm): 177.10, 168.76, 136.21, 133.48, 130.61, 123.52, 115.97, 48.23, 28.59.

**2.3.4. *N,N'*-Bis(5-Nitrosalicylidene)-1,7-Heptanediamine (L4)** (entry 4, Table 1). 5-nitrosalicylaldehyde: (0.668 g, 4 mmol); 1,7-heptanediamine: (0.26 g, 2 mmol); Yield: 88%; M.p: 150–152°C. Anal. Calcd.: C, 58.87; H, 5.65; N, 13.08. Found: C, 58.61; H, 5.78; N, 13.12. <sup>1</sup>H NMR, δ(ppm): 14.03–14.37 (s, 2H, O–H), 8.74 (s, 2H, CH=N), 8.68 (d, 2H, Ar–H), 8.27 (dd, 2H, Ar–H), 7.04 (d, 2H, Ar–H), 3.76 (t, 4H, CH<sub>2</sub>), 1.94 (m, 4H, CH<sub>2</sub>), 1.68 (m, 4H, CH<sub>2</sub>). <sup>13</sup>C NMR, δ(ppm): 180.25, 169.14, 136.24, 133.41, 130.62, 123.57, 115.94, 53.10, 26.57, 25.28.

**2.3.5. *N,N'*-Bis(5-Nitrosalicylidene)-1,8-Octanediamine (L5)** (entry 5, Table 1). 5-Nitrosalicylaldehyde: (0.668 g, 4 mmol); 1,8-octanediamine: (0.30 g, 2 mmol); yield: 85%; M.p: 161–164°C. Anal. Calcd.: C, 59.72; H, 5.92; N, 12.66. Found: C, 59.34; H, 5.66; N, 12.88. <sup>1</sup>H NMR, δ(ppm): 14.15–14.60 (s, 2H, O–H), 8.81 (s, 2H, CH=N), 8.68 (d, 2H, Ar–H), 8.27 (dd, 2H, Ar–H), 6.48 (d, 2H, Ar–H), 3.4 (t, 4H, CH<sub>2</sub>), 1.45 (m, 4H, CH<sub>2</sub>), 1.21 (m, 8H, CH<sub>2</sub>). <sup>13</sup>C NMR, δ(ppm): 176.24, 168.54, 135.23, 133.71, 130.7, 124.22, 115.41, 53.85, 30.73, 29.95, 27.52.

**2.3.6. *N,N'*-Bis(5-Nitrosalicylidene)-1,2-Phenylenediamine (L6)** (entry 6, Table 1). 5-Nitrosalicylaldehyde: (0.668 g, 4 mmol); 1,2-phenylenediamine: (0.22 g, 2 mmol); yield: 92%; M.p: 261–264°C. Anal. Calcd.: C, 59.12; H, 3.47; N, 13.79. Found: C, 58.94; H, 3.51; N, 13.66. <sup>1</sup>H NMR, δ(ppm): 14.02–14.60 (s, 2H, O–H), 8.81 (s, 2H, CH=N), 8.48 (d, 2H,

Ar-H), 7.94 (dd, 2H, Ar-H), 7.35 (s, 2H, Ar-H), 6.8 (d, 2H, Ar-H).  $^{13}\text{C}$  NMR,  $\delta(\text{ppm})$ : 175.4, 166.7, 139.1, 135.2, 133.2, 131.6, 128.2, 123.1, 120.8, 115.6.

2.3.7. *N,N'*-Bis(5-Nitrosalicylidene)-4,4'-Diaminodiphenylmethane (**L7**) (entry 7, Table 1). 5-Nitrosalicylaldehyde: (0.668 g, 4 mmol); 4,4'-diaminodiphenyl-methane: (0.40 g, 2 mmol); yield: 92%; M.p: 193–195°C. Anal. Calcd.: C, 65.32; H, 4.06; N, 11.28. Found: C, 65.23; H, 3.88; N, 11.65.  $^1\text{H}$  NMR,  $\delta(\text{ppm})$ : 13.55–14.08 (br, 2H, O-H), 8.87 (s, 2H, CH=N), 8.66 (d, 2H, Ar-H), 8.13 (dd, 2H, Ar-H), 6.63–7.48 (m, 10H, Ar-H), 2.65 (s, 2H,  $\text{CH}_2$ ); mass (EI): 498, 349, 195, 167, 108, 65, 39, 30.

2.3.8. *N,N'*-Bis(5-Nitrosalicylidene)-4,4'-Diaminodiphenylether (**L8**) (entry 8, Table 1). 5-Nitrosalicylaldehyde: (0.668 g, 4 mmol); 4,4'-diaminodiphenyl-ether: (0.40 g, 2 mmol); yield: 94%; M.p: 204–206°C. Anal. Calcd.: C, 62.65; H, 3.64; N, 11.24. Found: C, 62.54; H, 3.87; N, 11.58.  $^1\text{H}$  NMR,  $\delta(\text{ppm})$ : 14.15–14.58 (br, 2H, O-H), 8.70 (s, 2H, CH=N), 8.58 (d, 2H, Ar-H), 8.21 (dd, 2H, Ar-H), 6.57–7.31 (m, 10H, Ar-H); mass (EI): 498, 349, 195, 167, 108, 65, 39, 30.

2.4. Synthesis of Schiff Base Complexes of Ni(II) and Cu(II). Metal(II) acetate (1 mmol) were dissolved in 20 mL methanol as stirred for 20 minutes. Also, one mmol of selected Schiff base ligand is added to 20 mL of methanol in a 100 mL two-necked, round-bottomed flask that was provided with a reflux condenser and stirred to dissolve. The metal(II) salt solution was slowly added dropwise to the ligand solution with stirring, the resulting slurry was stirred under  $\text{N}_2$  at room temperature. After the end of the reaction time, the mixture is cooled until  $-5^\circ\text{C}$  over night, the microcrystalline solid was precipitated. The solution was filtered to eliminate excess unreacted metal acetate and the crude solids washed with ethanol ( $3 \times 10$  mL).

2.4.1. *N,N'*-1,2-Ethane-Bis(5-Nitrosalicylaldiminato)Nickel (II) **L1**[Ni(II)]. yield: 89%; M.p:  $>350^\circ\text{C}$ . Anal. Calcd.: C, 46.31; H, 2.91; N, 13.50. Found: C, 45.01; H, 3.05; N, 13.28.  $^1\text{H}$  NMR  $\delta(\text{ppm})$ : 9.01 (s, 2H, CH=N), 8.80 (d, 2H, Ar-H), 8.34 (dd, 2H, Ar-H), 6.77 (d, 2H, Ar-H), 3.6 (s, 4H,  $\text{CH}_2$ ).  $^{13}\text{C}$  NMR  $\delta(\text{ppm})$ : 170.47, 160.58, 140.22, 134.32, 129.38, 125.90, 114.64, 51.14.

2.4.2. *N,N'*-1,3-Propane-Bis(5-Nitrosalicylaldiminato)Nickel (II) **L2**[Ni(II)]. Yield: 83%; M.p:  $>350^\circ\text{C}$ . Anal. Calcd.: C, 47.59; H, 3.29; N, 13.06. Found: C, 47.05; H, 3.36; N, 12.66.  $^1\text{H}$  NMR  $\delta(\text{ppm})$ : 9.01 (s, 2H, CH=N), 8.80 (d, 2H, Ar-H), 8.34 (dd, 2H, Ar-H), 6.77 (d, 2H, Ar-H), 3.6 (t, 4H,  $\text{CH}_2$ ), 2.3 (q, 2H,  $\text{CH}_2$ ).  $^{13}\text{C}$  NMR  $\delta(\text{ppm})$ : 170.47, 160.58, 140.22, 134.32, 129.38, 125.90, 114.64, 51.14, 31.25.

2.4.3. *N,N'*-1,4-Butane-Bis(5-Nitrosalicylaldiminato)Nickel (II) **L3**[Ni(II)]. Yield: 90%; M.p:  $>350^\circ\text{C}$ . Anal. Calcd.: C, 48.80; H, 3.64; N, 12.65. Found: C, 48.30; H, 3.52; N, 12.31.  $^1\text{H}$  NMR,  $\delta(\text{ppm})$ : 8.83 (s, 2H, CH=N), 8.50 (d, 2H, Ar-H), 8.21 (dd, 2H, Ar-H), 6.89 (d, 2H, Ar-H), 3.83 (t, 4H,  $\text{CH}_2$ ),

2.50 (q, 4H,  $\text{CH}_2$ );  $^{13}\text{C}$  NMR,  $\delta(\text{ppm})$ : 177.10, 168.76, 136.21, 133.48, 130.61, 123.52, 115.97, 48.23, 28.59.

2.4.4. *N,N'*-1,7-Heptane-Bis(5-Nitrosalicylaldiminato)Nickel (II) **L4**[Ni(II)]. Yield: 87%; M.p:  $307^\circ\text{C}$ . Anal. Calcd.: C, 51.99; H, 4.57; N, 11.55. Found: C, 52.11; H, 4.21; N, 11.81.  $^1\text{H}$ -NMR,  $\delta(\text{ppm})$ : 8.83 (s, 2H, CH=N), 8.48 (d, 2H, Ar-H), 8.19 (dd, 2H, Ar-H), 6.40 (d, 2H, Ar-H), 3.40 (t, 4H,  $\text{CH}_2$ ), 1.89 (m, 4H,  $\text{CH}_2$ ), 1.17 (m, 6H,  $\text{CH}_2$ );  $^{13}\text{C}$  NMR,  $\delta(\text{ppm})$ : 179.09, 168.41, 135.32, 133.83, 130.74, 124.15, 115.26, 53.77, 30.68, 29.59, 27.33. Mass (EI): 428, 411, 393, 310, 263, 179, 163, 133, 104, 78, 55, 30.

2.4.5. *N,N'*-1,8-Octane-Bis(5-Nitrosalicylaldiminato)Nickel (II) **L5**[Ni(II)]. Yield: 88%; M.p:  $312^\circ\text{C}$ . Anal. Calcd.: C, 52.94; H, 4.85; N, 11.22. Found: C, 53.10; H, 4.62; N, 11.64.  $^1\text{H}$  NMR,  $\delta(\text{ppm})$ : 8.81 (s, 2H, CH=N), 8.68 (d, 2H, Ar-H), 8.27 (dd, 2H, Ar-H), 6.48 (d, 2H, Ar-H), 3.4 (t, 4H,  $\text{CH}_2$ ), 1.45 (m, 4H,  $\text{CH}_2$ ), 1.21 (m, 8H,  $\text{CH}_2$ );  $^{13}\text{C}$  NMR,  $\delta(\text{ppm})$ : 176.24, 168.54, 135.23, 133.71, 130.7, 124.22, 115.41, 53.85, 30.73, 29.95, 27.52.

2.4.6. *N,N'*-1,2-Phenylene-Bis(5-Nitrosalicylaldiminato)Nickel(II) **L6**[Ni(II)]. Yield: 87%; M.p:  $>350^\circ\text{C}$ . Anal. Calcd.: C, 51.88; H, 2.61; N, 12.10. Found: C, 51.41; H, 2.32; N, 12.35.  $^1\text{H}$  NMR  $\delta(\text{ppm})$ : 8.75 (s, 2H, CH=N), 8.32 (d, 2H, Ar-H), 7.78 (dd, 2H, Ar-H), 7.21 (s, 2H, Ar-H), 6.51 (d, 2H, Ar-H).  $^{13}\text{C}$  NMR  $\delta(\text{ppm})$ : 168.4, 162.7, 134.1, 133.2, 129.2, 129.6, 127.2, 120.1, 118.8, 113.6.

2.4.7. *N,N'*-4,4'-Diphenylemethane-Bis(5-Nitrosalicylaldiminato)Nickel (II) **L7**[Ni(II)]. Yield: 85%; M.p:  $>350^\circ\text{C}$ . Anal. Calcd.: C, 58.63; H, 3.28; N, 10.13. Found: C, 58.25; H, 3.11; N, 10.36.  $^1\text{H}$  NMR  $\delta(\text{ppm})$ : 8.9 (s, 2H, CH=N), 8.7 (d, 2H, Ar-H), 8.0 (dd, 2H, Ar-H), 6.3–7.1 (m, 10H, Ar-H), 2.5 (s, 2H,  $\text{CH}_2$ ); Mass (EI): 496, 349, 195, 167, 108, 65, 39, 30.

2.4.8. *N,N'*-4,4'-Diphenylether-Bis(5-Nitrosalicylaldiminato)Nickel(II) **L8**[Ni(II)]. Yield: 83%; M.p:  $337^\circ\text{C}$ . Anal. Calcd.: C, 56.25; H, 2.90; N, 10.09. Found: C, 56.03; H, 2.65; N, 10.25.  $^1\text{H}$  NMR  $\delta(\text{ppm})$ : 8.71 (s, 2H, CH=N), 8.60 (d, 2H, Ar-H), 8.10 (dd, 2H, Ar-H), 6.60–7.50 (m, 10H, Ar-H).

2.4.9. *N,N'*-1,2-Ethane-Bis(5-Nitrosalicylaldiminato)Copper (II) **L1**[Cu(II)]. Yield: 88%; M.p:  $335^\circ\text{C}$ . Anal. Calcd.: C, 45.77; H, 2.88; N, 13.35. Found: C, 45.89; H, 2.36; N, 13.41.  $^1\text{H}$  NMR,  $\delta(\text{ppm})$ : 8.91 (s, 2H, CH=N), 8.58 (d, 2H, Ar-H), 8.17 (dd, 2H, Ar-H), 6.91 (d, 2H, Ar-H), 4.1 (s, 4H,  $\text{CH}_2$ ).

2.4.10. *N,N'*-1,3-Propane-Bis(5-Nitrosalicylaldiminato) Copper(II) **L2**[Cu(II)]. Yield: 88%; M.p:  $>350^\circ\text{C}$ . Anal. Calcd. C, 47.06; H, 3.25; N, 12.91. Found: C, 47.12; H, 3.41; N, 12.62.  $^1\text{H}$  NMR  $\delta(\text{ppm})$ : 8.85 (s, 2H, HC=N), 8.38 (d, 2H, Ar-H), 8.27 (dd, 2H, Ar-H), 6.41 (d, 2H, Ar-H), 3.41 (t, 4H,  $\text{CH}_2$ ), 2.20 (q, 2H,  $\text{CH}_2$ ).

2.4.11. *N,N'*-1,4-Butane-Bis(5-Nitrosalicylaldiminato)Copper (II) **L3**[Cu(II)]. Yield: 90%; M.p:  $255^\circ\text{C}$ . Anal. Calcd.: C,

TABLE 2: The results of the reaction for preparation of Schiff base complexes.

Entry	Ligand	Metal(II)	Product	Time (min)	Yield <sup>a</sup> (%)	M.p (°C)
1	L1	Ni(II)	L1[Ni(II)]	80	89	>350
2	L2	Ni(II)	L2[Ni(II)]	85	83	>350
3	L3	Ni(II)	L3[Ni(II)]	85	90	>350
4	L4	Ni(II)	L4[Ni(II)]	90	87	307
5	L5	Ni(II)	L5[Ni(II)]	90	88	312
6	L6	Ni(II)	L6[Ni(II)]	90	87	>350
7	L7	Ni(II)	L7[Ni(II)]	90	85	>350
8	L8	Ni(II)	L8[Ni(II)]	80	83	337
9	L1	Cu(II)	L1[Cu(II)]	75	88	335
10	L2	Cu(II)	L2[Cu(II)]	70	88	>350
11	L3	Cu(II)	L3[Cu(II)]	85	90	255
12	L4	Cu(II)	L4[Cu(II)]	80	89	320
13	L5	Cu(II)	L5[Cu(II)]	90	82	283
14	L6	Cu(II)	L6[Cu(II)]	95	90	>350
15	L7	Cu(II)	L7[Cu(II)]	95	85	302
16	L8	Cu(II)	L8[Cu(II)]	90	88	>350

<sup>a</sup>Isolated yields.

48.27; H, 3.60; N, 12.51. Found: C, 48.10; H, 3.22; N, 12.11. <sup>1</sup>H NMR,  $\delta$ (ppm): 8.68 (s, 2H, CH=N), 8.35 (d, 2H, Ar-H), 8.01 (dd, 2H, Ar-H), 6.65 (d, 2H, Ar-H), 3.36 (t, 4H, CH<sub>2</sub>), 2.32 (q, 4H, CH<sub>2</sub>).

2.4.12. *N,N'*-1,7-Heptane-Bis(5-Nitrosalicylaldiminato) Copper(II) L4[Cu(II)]. Yield: 89%; M.p: 320°C. Anal. Calcd.: C, 51.48; H, 4.53; N, 11.43. Found: C, 51.32; H, 4.56; N, 11.34. <sup>1</sup>H NMR,  $\delta$ (ppm): 8.83 (s, 2H, CH=N), 8.48 (d, 2H, Ar-H), 8.19 (dd, 2H, Ar-H), 6.40 (d, 2H, Ar-H), 3.40 (t, 4H, CH<sub>2</sub>), 1.89 (m, 4H, CH<sub>2</sub>), 1.17 (m, 6H, CH<sub>2</sub>).

2.4.13. *N,N'*-1,8-Octane-Bis(5-Nitrosalicylaldiminato)Copper (II) L5[Cu(II)]. Yield: 82%; M.p: 283°C. Anal. Calcd.: C, 52.43; H, 4.80; N, 11.12. Found: C, 53.69; H, 4.02; N, 11.87. <sup>1</sup>H NMR,  $\delta$ (ppm): 8.7 (s, 2H, CH=N), 8.5 (d, 2H, Ar-H), 8.1 (dd, 2H, Ar-H), 6.3 (d, 2H, Ar-H), 3.1 (t, 4H, CH<sub>2</sub>), 1.2 (m, 4H, CH<sub>2</sub>), 1.0 (m, 8H, CH<sub>2</sub>).

2.4.14. *N,N'*-1,2-Phenylene-Bis(5-Nitrosalicylaldiminato) Copper(II) L6[Cu(II)]. Yield: 90%; M.p: >350°C. Anal. Calcd.: C, 51.34; H, 2.59; N, 11.97. Found: C, 51.85; H, 2.33; N, 11.62. <sup>1</sup>H NMR,  $\delta$ (ppm): 8.81 (s, 2H, CH=N), 8.48 (d, 2H, Ar-H), 7.94 (dd, 2H, Ar-H), 7.35 (s, 2H, Ar-H), 6.8 (d, 2H, Ar-H).

2.4.15. *N,N'*-4,4'-Diphenylmethan-Bis(5-Nitrosalicylaldiminato)Copper(II) L7[Cu(II)]. Yield: 85%; M.p: 302°C. Anal. Calcd.: C, 58.12; H, 3.25; N, 10.04. Found: C, 57.88; H, 3.46; N, 9.81. <sup>1</sup>H NMR,  $\delta$ (ppm): 8.74 (s, 2H, CH=N), 8.78 (d, 2H, Ar-H), 8.27 (dd, 2H, Ar-H), 6.81 (d, 2H, Ar-H), 6.41(m, 10H, phenyl ring), 3.74 (s, 2H, CH<sub>2</sub>).

2.4.16. *N,N'*-4,4'-Diphenylether-Bis(5-Nitrosalicylaldiminato)Copper(II) L8[Cu(II)]. Yield: 88%; M.p: >350°C. Anal. Calcd.: C, 55.77; H, 2.88; N, 10.01. Found: C, 55.12; H, 2.65; N, 10.18. <sup>1</sup>H NMR,  $\delta$ (ppm): 8.87 (s, 2H, CH=N), 8.66 (d, 2H, Ar-H), 8.13 (dd, 2H, Ar-H), 6.63–7.48 (m, 10H, Ar-H).

### 3. Results and Discussion

In this research, firstly, 2 moles of 5-nitro-salicylaldehyde and 1 mole diamine were reacted together and the corresponding products L1–L8 were obtained under mild and easy conditions (Scheme 1).

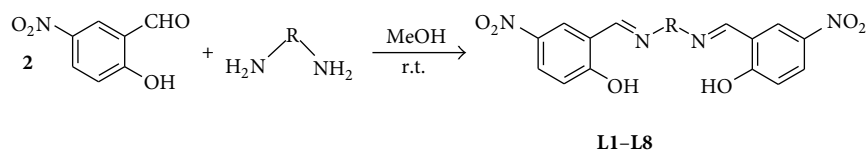
The confirmation of these products was demonstrated by spectroscopic and physical data. The results of these reactions were shown in Table 1. As indicated in this table, a lot of useful and convenient Schiff bases were afforded in high yields and appropriate reaction times.

In continuation of this research, in order to prepare all complexes, we applied Schiff base ligands in the reaction with equal moles amounts of nickel(II) and copper(II) acetate salts in methanol solution (Scheme 2).

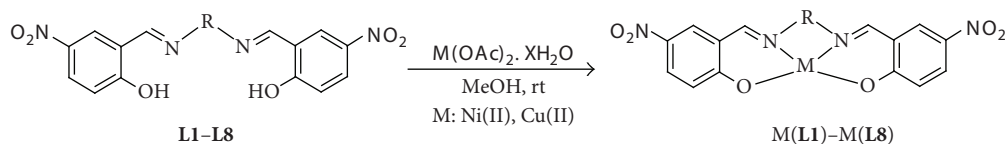
The corresponding results of these reactions were summarized in Table 2. Because of high reactivity regarding ligands in complex formation, the reactions have been proceeded under mild conditions at room temperature. This factor was restrained from oxidation of metal(II) to metal(III) in products.

The para-nitro-substitution of phenyl ring causes that the phenolic OH becomes more acidic; this affected on the conditions of direction synthesis of ligands and complexes. Basically, the aldehyde group becomes more active and increases the yield of the Schiff base formation. In addition to the formation of complexes, the phenolic hydrogen releases





SCHEME 1



SCHEME 2

easier and thus the complex formation occurs in low reaction time at room temperature.

**3.1. Electronic Spectra.** The electronic spectra of the ligands and their metal complexes were recorded in 1,2-dichloroethane. The electronic spectral data of the H2L ligands and their metal complexes are summarized in Table 3. The H2L shows two bands at 380–410 nm ( $\epsilon = 8.9 \times 10^3 \text{ mol}^{-1} \text{ cm}^{-1}$ ) and 275–315 nm ( $\epsilon = 7.7 \times 10^3 \text{ mol}^{-1} \text{ cm}^{-1}$ ), which may be assigned to the  $n \rightarrow \pi^*$  and  $\pi \rightarrow \pi^*$  transitions, respectively; the complexes show two bands in the 295–335 and 335–360 nm ranges which are assigned to intraligand transition [32–34]. In the complexes, the  $n \rightarrow \pi^*$  transitions due to the azomethine group are shifted to the lower energy. From these results, the nitrogen atom of the imines group appears to be coordinated to the metal ion [35]. The remainder of the observed bands at about 290–320 nm are assigned as  $\pi \rightarrow \pi^*$  type transitions involving molecular orbital located on the phenolic chromophore. The nickel(II) complexes show two bands at 430 and 581 nm and 430 and 570 nm are due to  ${}^3T_1 \rightarrow {}^3T_2$  transitions, indicating square planar environment around the nickel(II) ion [36–38]. For the nitro-substituted ligands, a considerable overlap between  $\pi \rightarrow \pi_2^*$  and  $\pi \rightarrow \pi_3^*$  is observed. The  $\pi \rightarrow \pi_1^*$  transition has been assumed to be localized mainly on the azomethine chromophore [39]. Instead, the  $\pi \rightarrow \pi_2^*$  band has been assigned to a transition involving mainly  $\pi$  molecular orbitals of the aromatic ring of the salicylideneimine moiety [40]. In the ligands, these bands were observed at about 280–320 nm. This blue shift in the complexes may be due to the electron's donation of a lone pair by the oxygen of the phenoxy group to the central metal atom.

**3.2. IR Spectra.** Important spectral bands of the H2L and its metal complexes are presented in Table 3. Significant frequencies were selected by comparing the IR spectra of the ligands with those of the metal complexes. The IR spectrum of the H2L shows broad medium bands in the 3450–3200 and 3100–2600  $\text{cm}^{-1}$  ranges, which attributed to intramolecular hydrogen bonding [41]. The spectra are showed broad strong bands at 3396 and 3250 and strong bands at 1662 and 1600  $\text{cm}^{-1}$  are assigned to the  $\nu(\text{NH})$ ,  $\nu(\text{C}-\text{O})$ , and

$\nu(\text{C}=\text{N})$ , respectively [42, 43]. Also, the strong and medium bands appear at 1570, 1508, and 1326  $\text{cm}^{-1}$ , correspond to  $\nu(\text{C}-\text{C})_{\text{Ar}}$ ,  $\nu(\text{CH}-\text{C})_{\text{AL}}$ , and  $\nu(\text{C}-\text{O})$ , respectively [44].

The ligands and complexes were characterized mainly using the imine and phenolic bands. The main infrared bands and their assignments are listed in Table 3. The IR spectra of the complexes in comparison with the free ligands to determine the changes that might have taken place during the complexation. The band at 1610–1640  $\text{cm}^{-1}$  is characteristic of the azomethine nitrogen atom in the free ligand. The observed lowering in this frequency to region 1590–1615  $\text{cm}^{-1}$  in all the complexes and indicates the involvement of the azomethine nitrogen atom in coordination with metalation [45, 46]. The spectra of the ligands shows broad bands in the rang 3200–3500  $\text{cm}^{-1}$  assignable to intramolecular H-bonded of phenolic groups [47], which are absent in the spectra of their complexes, indicating that the oxygen of the –OH groups is coordinated to the metal ion [48]. Thus, the entire ligands act as tetradentate chelating compound coordinated to metal ion through two oxygen and two nitrogen atoms [49]. These data are well in accordance with those of reported complexes [50, 51].

Upon coordination bands at 1512 and 1341  $\text{cm}^{-1}$  which are typical of nitro group, nitro ligands undergo minor changes in complexes. It may therefore be that the nitro group is not coordinated to the metal ions. In all the complexes, the bands at 617–461  $\text{cm}^{-1}$  and 461–420  $\text{cm}^{-1}$  rang can be attributed to the  $\nu_{\text{M}-\text{N}}$  and  $\nu_{\text{M}-\text{O}}$  modes, respectively.

**3.3. Magnetic Properties of Complexes.** Molecular paramagnetism is a characteristic property of unpaired electron systems. In most coordination compounds of the transition metals and some organometallic compounds as well, paramagnetic behavior is encountered due to the incompletely filled 3d, 4d, and 5d electron shell. It should be noted that simple paramagnetism will be found only if there is sufficient magnetic dilution. This is the case if, due to the presence of large organic ligands, the paramagnetic centers are well separated, thus avoiding cooperative interactions of the ferro and antiferromagnetic type. Magnetic susceptibility amounts of the complexes were summarized in Table 4.

TABLE 3: UV-Vis spectral data (DMF, nm) and IR bands ( $\text{cm}^{-1}$ ) of ligands and their complexes.

Entry	$n \rightarrow \pi^*$ (C=N)	$\pi \rightarrow \pi^*$ (rings)	d $\rightarrow$ d	$\nu(\text{C}=\text{N})$	$\nu(\text{Ph}-\text{O})$	$\nu(\text{O}-\text{H})$	$\nu(\text{NO}_2)$	$\nu(\text{M}-\text{N}, \text{M}-\text{O})$
L1	409	284	—	1635	1330	3360	1532, 1338	—
L1[Cu(II)]	418	288	568	1608	1335	—	1530, 1341	460–490
L1[Ni(II)]	427	275	574	1615	1332	—	1539, 1347	465–492
L2	410	287	—	1643	1325	3353	1530, 1342	—
L2[Cu(II)]	429	280	570	1617	1338	—	1534, 1352	464–490
L2[Ni(II)]	430	271	571	1602	1339	—	1537, 1350	469–484
L3	407	291	—	1634	1324	3330	1534, 1329	—
L3[Cu(II)]	420	280	588	1603	1330	—	1541, 1323	451–480
L3[Ni(II)]	435	284	573	1609	1328	—	1551, 1319	455–487
L4	417	287	—	1641	1338	3382	1539, 1327	—
L4[Cu(II)]	427	279	582	1618	1324	—	1548, 1336	449–490
L4[Ni(II)]	432	283	574	1612	1329	—	1553, 1320	460–485
L5	416	295	—	1647	1321	3340	1527, 1355	—
L5[Cu(II)]	419	285	560	1615	1329	—	1529, 1353	458–494
L5[Ni(II)]	432	282	572	1600	1334	—	1527, 1349	462–481
L6	421	298	—	1642	1327	3294	1531, 1357	—
L6[Cu(II)]	428	284	561	1603	1338	—	1529, 1352	459–495
L6[Ni(II)]	430	279	570	1607	1336	—	1530, 1354	460–490
L7	410	289	—	1631	1319	3285	1538, 1340	—
L7[Cu(II)]	425	285	584	1615	1307	—	1541, 1348	465–498
L7[Ni(II)]	433	300	575	1620	1310	—	1530, 1339	458–487
L8	414	308	—	1645	1322	3354	1538, 1356	—
L8[Cu(II)]	426	302	587	1614	1335	—	1537, 1359	462–487
L8[Ni(II)]	431	294	568	1611	1331	—	1530, 1352	454–489

TABLE 4: Magnetic susceptibility amounts of complexes.

Entry	Complexes	$\chi_m (\times 10^{-4})/\text{emu}/\text{gr.Oe}$
1	L1[Ni(II)]	2.2419
2	L2[Ni(II)]	2.1158
3	L3[Ni(II)]	2.0852
4	L4[Ni(II)]	2.0814
5	L5[Ni(II)]	2.0805
6	L6[Ni(II)]	2.2852
7	L7[Ni(II)]	2.2046
8	L8[Ni(II)]	2.1910
9	L1[Cu(II)]	2.3014
10	L2[Cu(II)]	2.2710
11	L3[Cu(II)]	2.2511
12	L4[Cu(II)]	2.2020
13	L5[Cu(II)]	2.1012
14	L6[Cu(II)]	2.8852
15	L7[Cu(II)]	2.3019
16	L8[Cu(II)]	2.2546

3.4.  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR Spectra. Spectra of all the complexes were recorded in DMSO- $d_6$  solution at 400 MHz

and chemical shifts are in units of ppm relative to TMS as internal standard on the delta ( $\delta$ ) scale. The general  $^1\text{H}$  NMR spectrum of the Schiff bases in DMSO shows the following signals: 14.0–14.5  $\delta$  a singlet and broad band for phenolic (O–H) group, 8.9  $\delta$  a singlet band for azomethine hydrogen (HC=N), 8.5–8.6  $\delta$  a doublet band for Ar–H, 8.1–8.2  $\delta$  as dd for Ar–H and 6.9–7.1 ppm a doublet peak for Ar–H. The absence of peak in 14.0 ppm, noted in the metal(II) complex, indicates the loss of the –OH proton due to complexation [52].

Intermolecular hydrogen bonding also accounts for the high frequency of the signals for the orthophenolic hydrogens in all the Schiff bases. By comparing the  $^1\text{H}$  NMR spectra of all the Schiff bases with those of their corresponding metal(II) complexes, it is noted that there is a downfield shift in the frequency of azomethine protons of the aromatic bridge and up to field shift in the aliphatic bridge confirming coordination of the metal ion to both groups.

#### 4. Conclusion

In this study, we have reported a mild and convenient method for synthesis of some Schiff base complexes of metal(II) at room temperature. Also the desired Schiff bases for preparation of these complexes have been obtained through

easy, simple, and efficient reaction of nitrosalicylaldehyde with various diamines. The resulting products have been afforded in excellent yields and efficient reaction times. The ligands are found to be bound to the metal atom through the oxygen atoms of the hydroxyl groups and nitrogen atoms of imine chromophore which is also supported by spectroscopic techniques.

## Acknowledgment

The authors gratefully acknowledge partial support of this work by the Research Affairs Office of the University of Kashan, Iran.

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