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Some Transition Metal Complexes of NO Type Schiff Base: Preparation and Characterization

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Abstract. Metal complexes of Schiff base ligand (5-bromo-3-methoxysalicyliden-p-iminoacetophenone oxime) derived from 5-bromo-3-methoxsalicylaldehyde and p-aminoacetophenoneoxime is reported. Schiff base was found to be bidentate ligand involving the imino nitrogen and carboxyl oxygen atoms in the complexes. Metal to ligand ratio were found to be 1:2 for all of the complexes. Co(II), Ni(II), and Zn(II) complexes have been found tetrahedral geometry and Cu(II) complex has been found four coordinated geometry. The complexes are found to have the formulae [M(L)2]. The compounds obtained have been characterized by their elemental analyses, IR, 1H-NMR, 13C-NMR, UV spectra, magnetic susceptibility and thermogravimetric analyses (TGA).

Keywords: Schiff base, transition metal complexes

N,O Tipi Schiff Bazının Bazı Geçiş Metal Kompleksleri: Hazırlanması ve Karakterizasyonu

Özet. Bu çalışmada, 5-brom-3-metoksi salisilaldehit ve p-aminoasetofenon oksimden türetilmiş Schiff bazı ligandı (5-brom-3-metoksisalisiliden-p-iminoasetofenon oksim) nın metal kompleksleri rapor edildi. Bütün komplekslerde Schiff bazlarının metal iyonuna imin azotu ve fenolik oksijeninden bağlanarak iki dişli şelat olarak davrandığı ve yine bütün komplekslerde M:L oranının 1:2 olduğu görüldü. Co+2, Ni+2 ve Zn+2 komplekslerinin tetrahedral ve Cu+2 kompleksinin ise dört koordinasyonlu yapıya sahip olduğu belirlendi. Komplekslerin [M(L)2] formülüne sahip oldukları bulunmuştur. Elde edilen bileşiklerin yapıları, elementel analiz, IR, 1H-NMR, 13C-NMR, Uv, manyetik süsseptibilite ve termogravimetrik analiz ile karakterize edilmiştir.

Anahtar Kelimeler: Schiff bazı, geçiş metal kompleksleri

1. INTRODUCTION

Schiff bases [1-6] are useful chelators because of their ease of preparation, structural varieties, varied denticities and subtle steric and electronic control on their framework. Recently a great deal of interest have developed in the synthesis and characterization of transition metal complexes containing Schiff bases as ligands due to their applications as catalysts for many reactions and relation to synthetic and natural oxygen carriers.

Schiff base compounds are readily synthesized from the condensation reaction between aldehydes and primary amines, which have been widely investigated for their biological activities, such as antibacterial, antifungal, and antitumor [7-11]. The imine N atom and some other donor atoms of the Schiff base compounds easily coordinate to metal atoms, forming versatile complexes [12-14].

We now report the synthesis and characterization of new tetrahedral Co(II), Ni(Il), Cu(II) and Zn(II) complexes with 5-bromo-3-methoxysalicyliden-*p*-iminoacetophenone oxime (LH), as well as general comparison of a tendency for tetra-coordinated structure formation in the case of d^7, d^8, d^9 and d^{10} configurations. The Schiff base ligand used in this study is shown in Scheme 1. All compounds were

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characterized by elemental analyses, IR, ^{1}H-NMR, ^{13}C-NMR spectra, UV spectra, magnetic susceptibility measurements and thermogravimetric analyses (TGA).

2. EXPERIMENTAL

Elemental analyses (C, H, N) were carried out using LECO-932 CHNSO by Technical and Scientific Research Council of Turkey (TUBITAK). IR spectra were recorded on a Mattson 1000 FT-IR spectrometer using KBr pellets. ^{1}H and ^{13}C-NMR spectra were taken from a Bruker GmbH Dpx-400 MHz High Performance Digital FT-NMR spectrometer. Electronic spectra were obtained on a Shimadzu 1700 UV spectrometer. Magnetic susceptibilities were measured by using 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 constant. TGA curves were recorded on a Shimadzu TG-50 thermo balance.

2.1. 5-bromo-3-methoxysalicyliden-*p*-iminoacetophenone oxime (LH)

A solution of *p*-aminoacetophenoneoxime (1.50 g, 10 mmol) dissolved in 15 ml absolute ethanol was added dropwise to 5-bromo-3-methoxysalicylaldehyde (2.31 g, 10 mmol) and p-toluene sulfonic acid (0.01 g) dissolved in 35 ml absolute ethanol under reflux at 60 °C with continuous stirring for 3 h. Precipitate was filtered off after overnight, washed several times with hot water, cold ethanol and diethyl ether and finaly dried in vacuum.

IR spectrum (υ, cm^{-1}): 3400 (oxime O-H), 3240 (phenolic O-H), 1625 (phenolic C=N), 1600 (oxime C=N), 1270 (C-O), 1005 (N-O); ^{1}H-NMR ($CDCl_3$-DMSO-*d*$_6$, δ, ppm): 14.00 (s, 1H, phenolic O$\underline{H}$), 11.00 (s, 1H, oxime O$\underline{H}$), 9.39 (s, 1H, azomethine C$\underline{H}$=N), 7.68-6.82 (m, 6H aromatic $\underline{H}$,), 3.74 (s, 3H, OC$\underline{H}_3$), 1.90 (s, 3H, C$\underline{H}_3$); ^{13}C-NMR ($CDCl_3$-DMSO-*d*$_6$, δ, ppm): 164.58 (oxime $\underline{C}$=NOH), 163.69 ($\underline{C}$H=N), 162.40 (phenolic $\underline{C}$-OH), 156.12-116.80 (aromatic $\underline{C}$), 56.30 (O$\underline{C}H_3$), (21.81 ($\underline{C}H_3$).

2.2. Bis (*p*-aminoacetophenoneoxime-5-bromo-3-methoxysalicylaldiminato) cobalt(II) Co(L)$_2$

A sample of the ligand (0.36 g, 1.00 mmole) was dissolved in absolute ethanol (25 mL). A solution of $Co(AcO)_2.4H_2O$ (0.13 g, 0.50 mmole) in 15 mL absolute ethanol was added dropwise to the ligand solution with continuous stirring at 50 ^{0}C for 14 h. The precipitated complex was filtered off after overnight, washed with hot H_2O, cold EtOH and cold Et_2O several times and dried in vacuo.

IR spectrum (υ, cm^{-1}): 3300 (oxime O-H), 1610 (phenolic C=N), 1599 (oxime C=N), 1290 (C-O), 1006 (N-O).

2.3. Bis (*p*aminoacetophenoneoxime-5-bromo-3-methoxysalicylaldiminato) nickel(II) Ni(L)$_2$

Ni(L)$_2$was prepared following the same procedure as described for Co(L)$_2$, starting with LH (0.36 g, 1.00 mmole) and $Ni(AcO)_2.4H_2O$ (0.12 g, 0.50 mmole).

IR spectrum (υ, cm^{-1}): 3310 (oxime O-H), 1615 (phenolic C=N), 1600 (oxime C=N), 1300 (C-O), 1006 (N-O).

2.4. Bis (*p*aminoacetophenoneoxime-5-bromo-3-methoxysalicylaldiminato) copper(II) Cu(L)$_2$

Cu(L)$_2$was prepared following the same procedure as described for Co(L)$_2$, starting with LH (0.36 g, 1.00 mmole) and $Cu(AcO)_2.H_2O$ (0.13 g, 0,50 mmole).

IR spectrum (υ, cm^{-1}): 3290 (oxime O-H), 1610 (phenolic C=N), 1600 (oxime C=N), 1310 (C-O), 1006 (N-O).

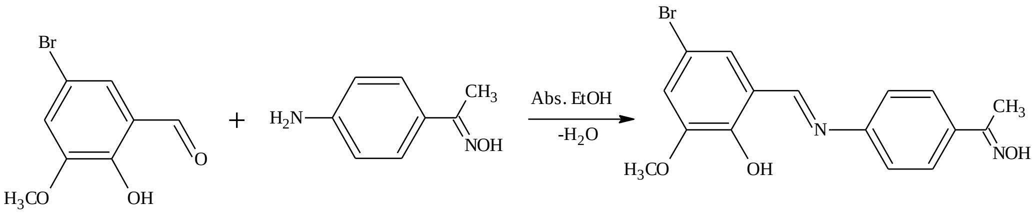
2.5. Bis(*p*aminoacetophenoneoxime-5-bromo-3-methoxysalicylaldiminato) zinc(II) Zn(L)$_2$

Zn(L)$_2$ was prepared following the same procedure as described for Co(L)$_2$, starting with LH (0.36 g, 1.00 mmole) and $Zn(AcO)_2.2H_2O$ (0,11 g, 0,50 mmole).

IR spectrum (υ, cm^{-1}): 3325 (oxime O-H), 1605 (phenolic C=N), 1599 (oxime C=N), 1295 (C-O), 1006 (N-O); ^{1}H-NMR ($CDCl_3$-DMSO-d_6, δ, ppm): 10.99 (s, 2H, oxime O$\underline{H}$), 9.13 (s, 2H, azomethine C$\underline{H}$=N), 7.70-6.80 (m, 12 H aromatic $\underline{H}$,), 3.74 (s, 6H, OC$\underline{H}_3$), 1.90 (s, 6H, C$\underline{H}_3$); ^{13}C-NMR ($CDCl_3$-DMSO-d_6, δ, ppm): 164.59 (oxime $\underline{C}$=NOH), 165.95 ($\underline{C}$H=N), 165.63 (phenolic $\underline{C}$-OH), 156.10-116.80 (aromatic $\underline{C}$), 56.30 (O$\underline{C}H_3$), 21.82 ($\underline{C}H_3$).

3. RESULTS AND DISCUSSION

The ligand (LH) was prepared by reacting equimolar amounts of 5-bromo-3-methoxysalicylaldehyde with p-aminoacetophenoneoxime in absolute ethanol (scheme 1). The structures of the ligand and the complexes were established from their IR, ^{1}H- and ^{13}C-NMR spectra, UV spectra, elemental analyses, magnetic susceptibility measurements and thermogravimetric analyses.



Scheme 1. Structure of the ligand.

The general equations for the formation of the complexes are shown below:

$2LH + Co(CH_3COO)_2 \cdot 4H_2O \rightarrow Co(L)_2 + 2CH_3COOH + 4H_2O$

$2LH + Ni(CH_3COO)_2 \cdot 4H_2O \rightarrow Ni(L)_2 + 2CH_3COOH + 4H_2O$

$2LH + Cu(CH_3COO)_2 \cdot H_2O \rightarrow Cu(L)_2 + 2CH_3COOH + H_2O$

$2LH + Zn(CH_3COO)_2 \cdot 2H_2O \rightarrow Zn(L)_2 + 2CH_3COOH + 2H_2O$

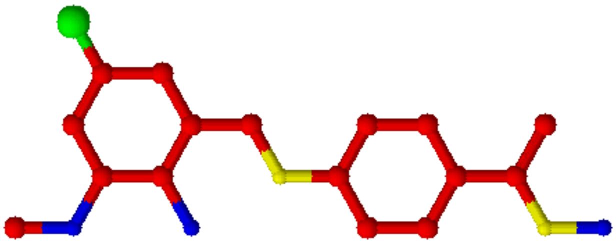
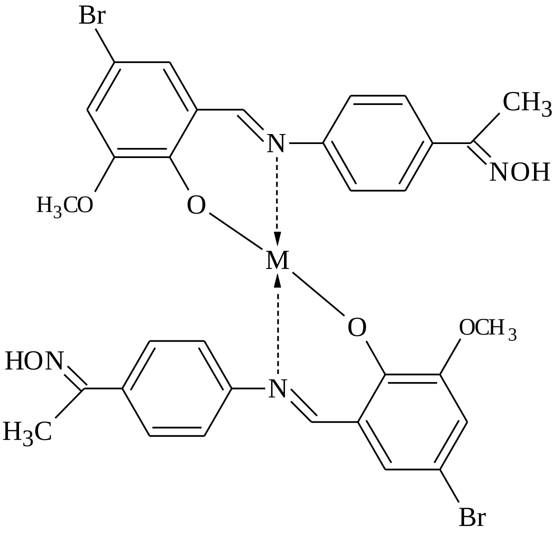


Figure 1. Conformation of ligand.

All of the metal complexes have the composition $M(L)_2$ where L is 5-bromo-3-methoxysalicyliden-p-aminoacetophenoneoxime. The analytical data are in good agreement with the proposed general molecular formula (Figure 2).



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

Figure 2. Suggested structure of the tetrahedral complexes of ligand.

3.1. Infrared Spectra

The infrared spectrum of LH shows bands at *ca.* 3400, 3240, 1625, 1600, 1270 and 1005 cm^{-1}, assigned to O-H (oxime) [15-17], O-H (phenolic), C=N (azomethine), C=N (oxime) [18-20], C-O and N-O, respectively. These values are in agreement with those found for similar compounds [21,22]. The infrared spectral bands which are most useful for the determination of the mode of coordination of the ligand are listed in experimental section. The spectra of all metal complexes show no absorption band corresponding to O-H (phenolic) of the aldehyde moiety. A strong band is observed in the free ligand at 1625 cm^{-1}, characteristic of the azomethine (C=N) group [23]. Coordination of the Schiff base to the cobalt, nickel, copper and zinc ion through the azomethine nitrogen atom is expected to reduce the electron density in the azomethine link and thus lower the (C=N) absorption frequency. Hence, this band undergoes a shift to lower frequency to 1605-1615 cm^{-1} after complexation indicating coordination of the azomethine nitrogen [24] to cobalt, nickel, copper and zinc. A medium intensity band at 3240 cm^{-1} in the spectra of the free ligand due to (O-H) was absent in the spectra of all the complexes, indicating the deprotonation of Schiff base prior the coordination [25,26] through its oxygen atom. This is further supported by an increase in the absorption frequency of the phenolic C-O band from 1270 cm^{-1} in the spectra of the free ligand to 1290-1310 cm^{-1} in the spectra of the complexes, indicating that the other coordination site of the Schiff base is the phenolic oxygen [27]. The practically unchanged O-H at 3400 and C=N at 1600 cm^{-1} point that oxime group does not coordinate to metal atoms by neither oxygen nor nitrogen atoms.

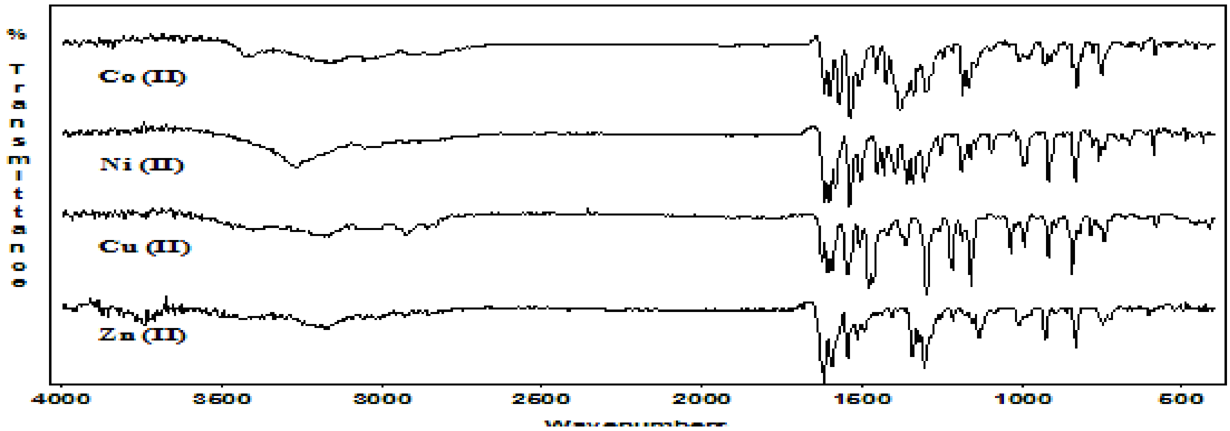


Figure 3. IR spectrum of complexes.

The NMR spectra of the Schiff base and its diamagnetic zinc(II) complex was recorded in $CDCl_3$/DMSO-d_6. ^{1}H- and ^{13}C-NMR assignments are detailed in experimental section. The Schiff base exhibited signals due to all the expected protons in their expected region and have been found from the integration curve equivalent to the total number of protons deduced from the proposed structure. These were compared with the reported [28] signals of the known comparable compounds and give further support for the compositions of the new ligand as well as their complexes, also suggested by their IR and elemental analyses data.

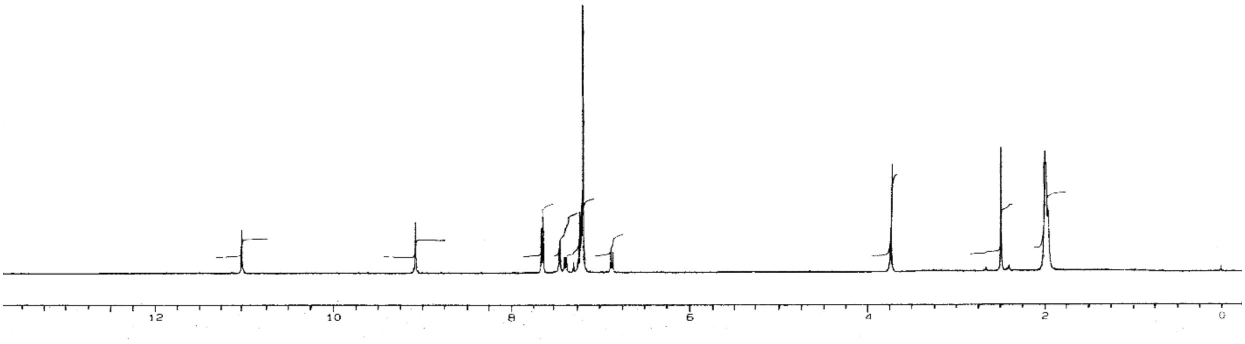


Figure 4. ^{1}H-NMR spectrum of Zn (II) complex.

Characteristic ^{1}H-NMR peaks are at 14.00 (phenolic OH), 11.00 (oxime OH) [16,22,28], 9.39 (azomethine CH=N), 6.82-7.68 (Arom-H) and 1.90 ppm (CH_3). The phenolic OH signal at 14.00 ppm disappeared upon addition of D_2O to the solution, indicating that it is an acidic proton. As can be seen in the ^{1}H-NMR spectra of zinc(II) complex there is no OH peaks expected. The signal due to azomethine group underwent an up field shift of 0.26 ppm in the complex indicating coordination of this group with the Zn(II) ion. The absence of the phenolic (O-H) proton signal in the zinc(II) complex indicates the coordination by phenolic oxygen to the metal ion after deprotonation [29-31]. More detailed information about the structure of ligand was provided by ^{13}C-NMR spectral data CH=N and C-OH carbon atoms are observed at 163.69 and 162.40 ppm respectively for Schiff base. ^{13}C-NMR spectrum of zinc(II) complex CH=N and C-OH carbon atoms are observed at 165.95 and 165.63 ppm respectively. The rest of carbon atoms, likewise showed similar diagnostic features for the free ligand as well as its complex as expected. The signals corresponding to the O-H proton and C=N-OH carbon, (both in oxime 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.

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

Compounds	Formula	F.W (g/mol)	Color	Yield (%)	μ_{eff} (B.M.)	Elemental analysis Calculated (found) (%)		
						C	H	N
LH	$C_{16}H_{15}BrN_2O_3$	363.21	yellow	80	—	52.91 (53.30)	4.16 (3.79)	7.71 (8.08)
$Co(L)_2$	$CoC_{32}H_{28}Br_2N_4O_6$	783.33	brown	55	4.15	49.07 (48.71)	3.60 (3.33)	7.15 (6.85)
$Ni(L)_2$	$NiC_{32}H_{28}Br_2N_4O_6$	783.09	green	59	2.91	49.08 (48.77)	3.60 (3.36)	7.15 (6.80)
$Cu(L)_2$	$CuC_{32}H_{28}Br_2N_4O_6$	787.94	light red	51	1.83	48.78 (49.16)	3.60 (3.20)	7.15 (6.76)
$Zn(L)_2$	$ZnC_{32}H_{28}Br_2N_4O_6$	789.80	orange	63	dia	48.66 (49.01)	3.57 (3.97)	7.09 (6.74)

The general characteristic properties of the complexes and the Schiff base are shown in Table 1. The elemental analysis results agree with the calculated values showing that the complexes have 1:2 metal/ligand ratios. The elemental analysis confirmed the compositions of the above synthesized compounds.

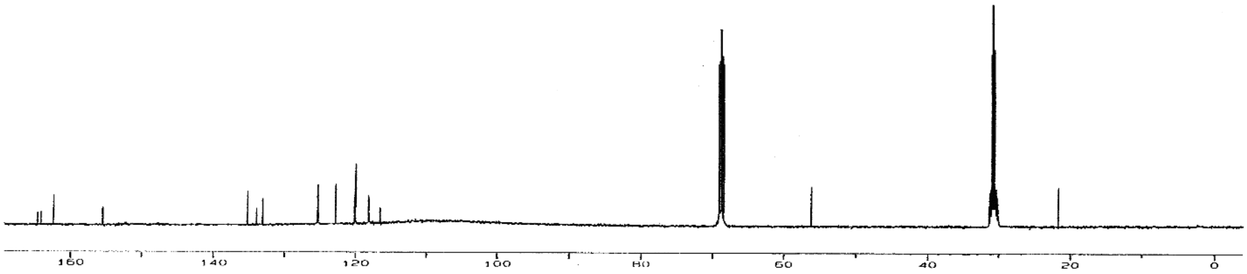


Figure 5. ^{13}C-NMR spectrum of Zn (II) complex.

The room temperature magnetic moment values of the complexes are given in Table 1. The magnetic moment value of 4.15 B.M. measured for the cobalt complex lies in the range expected for a d^7 system, which contains one unpaired electron with tetrahedral geometry [32]. The measured value of 2.91 B.M. for the nickel complex suggests tetrahedral geometry for this complex [33]. The observed magnetic moment of 1.83 B.M. is consistent with a four coordinated copper(II) complex [34]. The zinc(II) complex was found to be diamagnetic as expected. The electronic spectra of the Schiff base and all the complexes were recorded in DMF at room temperature. The electronic spectral data of the ligand and its complexes are given in experimental section. The aromatic band of the ligand at 275 nm is attributed to benzene $\pi \rightarrow \pi^*$ transition. The band around 390 nm is due to the $n \rightarrow \pi^*$ transition of the non-bonding electrons present on nitrogen of the azomethine group in the Schiff base. The complexes of cobalt(II), nickel(II) and copper(II) show less intense shoulders at *ca.* 560-670 nm (ε = 155-190 L mol^{-1} cm^{-1}), which are assigned as *d-d* transition of the metal ions. The former band is probably due to the $^4A_2 \rightarrow ^4T_1$ (P) for Co(II), $^3A_2 \rightarrow ^3T_2$ (F) for Ni(II) and $^2T_2 \rightarrow ^2E$ (G) for Cu(II) transition of tetrahedral geometry. All the complexes show an intense band at *ca* 370-394 nm which is assigned to $n \rightarrow \pi^*$ transition associated with azomethine linkage. The spectra of all the complexes show intense band at *ca* 425-440 nm (ε = 1.50-4.00 X 10^3 L mol^{-1} cm"^{1}), which can be assigned to charge transfer transition of tetrahedral geometry [35-37].

The thermogravimetric (TG) curves for the complexes were obtained at a heating rate of 10°C/min and a 30 mL/min flowing nitrogen over a temperature range of 20-800°C. Approximately 10 mg samples of the complexes were used in each case. The TG curves showed that the thermal decomposition of the complexes takes place in two steps. Furthermore, it is known that the electronegativity and the atomic radius of the central metal atom also affect the thermal stability [38]. Thermogravimetric studies of all the complexes showed no weight loss up to 205 °C indicating absence of water in the complexes. The inflation of the TG curves of all the complexes at a temperature under 720 °C indicates the decomposition of the fully organic part of the chelate, leaving metallic oxide at the final temperature [39,40]. The thermal stability of all complexes increases in the order: Cu < Zn < Ni < Co.

4. CONCLUSION

Our group has been heavily engaged in synthesis of substituted oximes and their Schiff-base derivatives. Many Schiff-base derivatives, containing substituted oximes, were synthesized, characterized in detail and used for complexation with some transition metal salts. Functional groups, such as oxime, on the complexes have no effect. These functional groups are very far from the pendants taking part in the complexation. The Schiff-base ligand and its Co(II), Ni(II), Cu(II) and Zn(II) metal complexes were synthesized and characterized by elemental analyses, IR, ^{1}H- and ^{13}C-NMR spectra, Uv spectra, magnetic susceptibility measurements and thermogravimetric analyses (TGA). All complexes are mononuclear and tetrahedral. According to results obtained from TGA, IR and elemental analyses, there are no lattice/coordinated water molecules in the complexes. For these complexes, additional analytical and physical data are given in Table 1. The suggested modes of coordination are shown in Figure 2.

Acknowledgements

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