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The Synthesis and Characterization of Some Metal Complexes Derived From 1,3-Dioxalane Groups and Vic-Dioxime Ligands

Sibel Duman[1] Memet Şekerci[2]

[1]Department of Chemistry, Faculty of Arts and Sciences, Bingol University, Bingol, TURKEY
[2]Department of Chemistry, Faculty of Arts and Sciences, Firat University, Elazig, TURKEY

Corresponding author
Email: sibelduman23@hotmail.com

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Abstract

A novel substituted vic-dioxime ligand containing the amino group, N,N′-[3,3′-{ethane-1,2-di-yl-bis(oxy)bis(3,1-phenylene)} bis(N′-hydroxy)-2-(hydroxyimino)acetimid amide], L^1, has been prepared from 1,2-bis(m-aminophenoxy)ethane, anti-chloroglyoxime. This vic-dioxime ligand with Cu(II), Ni(II), Co(II) and Cd(II) complexes of 6,7-O-Cyclopentlydine-1-amino-4-azaheptane, L^2, have been reacted at 60-65 °C temperature. The ligand^1:metal:ligand^2 ratio of these complexes have been found to be 1:2:2. The Cu(II), Ni(II), Co(II) and Cd(II) complexes of these ligands are proposed to be octahedral. The vic-dioxime (L^1) with some transition metal complexes of amine (L^2) have been characterized by elemental analyses, molar conductivity, magnetic susceptibility, IR, ^{1}H-NMR, UV-Vis spectra and TGA techniques.

Keywords: Vic-dioxime, Anti-chloroglyoxime, Transition Metal Complexes.

INTRODUCTION

Oximes and their metal complexes are of current interest due to their rich physicochemical properties, reactivity patterns and potential applications in many important chemical processes in the areas of medicine, bioorganic system, and catalysis, electrochemical and electrooptical sensors [1-4]. The presences of mildly acidic hydroxyl groups and slightly basic nitrogen atoms makes vic-glyoximes amphoteric ligands which from corrin-type square planar, square-pyramidal and octahedral complexes with metal ions such as nickel(II), cupper(II), cobalt(II) and cobalt(III) as central atoms [5,6]. The high stability of the complexes prepared with vic-dioxime ligands has been extensively used for purposes including model compounds for vitamin B_{12} or trace metal analysis [7,8].

Vic-dioximes and their complexes constitute an important class of compounds having versatile reactivities [9]. Oxime metal chelates are biologically active and are reported to posses semiconducting properties [10,11]. The substitution of the vic-dioxime moiety affects the structure and stability of the complex [12]. Compounds containing the 1,3-dioxalane groups are used as solvents, additive compounds, and corrosion retardants, while polymers containing 1,3-dioxalane groups exhibit semiconducting behavior [13,14].

In our previous studies we investigated the synthesis and characterization of various transition metal complexes of novel bidentate compounds [15,16]. In the present study, new amino vic-dioxime complexes derived from N,N′-[3,3′-{ethane-1,2-di-yl-bis(oxy) bis(3,1-phenylene)}bis(N′-hydroxy)-2-(hydroxyimino) acetimidamide] (L^1) with Cu(II), Ni(II), Co(II) and Cd(II) complexes of 6,7-O-cyclopentilydine-1-amino-4-azaheptane (L^2) used as metal salts have been prepared and characterized by electronic, infrared, molar conductivity, magnetic and ^{1}H-NMR spectral measurements in addition to elemental and thermogravimetrical analyses. The data revealed same geometries around the metal ions, depending on both the ligand and metal ions.

MATERIALS and METHODS

A new vic-dioxime ligand, N,N′-[3,3′-{ethane-1,2-di-yl-bis(oxy)bis(3,1-phenylene)}bis(N′-hydroxy)-2-(hydroxyimino)acetimidamide] (L^1), derived from reaction of 1,2-bis(m-aminophenoxy)ethane [17,18] and *anti*-chloroglyoxime [19], 6,7-O-cyclopentilydine-1-amino-4-azaheptane (L^2) [14,20,21] and Cu(II), Ni(II), Co(II) and Cd(II) complexes of the L^2 [14,20,21] were synthesized and characterized as described in the literature. All the reagents used were purchased from Merck, Across or Lakbim company and chemically pure.

Elemental analyses (C, H, N) were performed on a LECO-932 CHNS-O elemental analyses apparatus. IR spectra were recorded on a Perkin Elmer Precisely Spectrum One Spectrometer as KBR pellets. ^{1}H-NMR spectra were recorded on a Bruker GmbH DPX-300 MHz High Performance Digital FT-NMR spectrometers (in DMSO-d_6). Electronic spectra were obtained on a Shimadzu UV-1700 spectrometer. Magnetic susceptibilities were determined on a Sherwood Scientific Magnetic Susceptibility balance (Model MK1) at room temperature using $Hg[Co(SCN)_4]$ as a calibrate; diamagnetic corrections were calculated from Pascal's constants [22]. Molar conductances were measured on a CMD 750WPA conductometer [23]. TGA curve was recorded on a Shimadzu DTG-60AH thermo balance.

Synthesis of L^1 with Cu(II) Ni(II), Co(II) and Cd(II) complexes of (L^2)

The solution of $[ML^2(AcO)_2]\cdot nH_2O$ where M is Cu(II) and Cd(II), n is 0 and 2, respectively, (0.40 g for Cu(II), 0.48 g for Cd(II), 1.0 mmol) and the solution of $[M_2L^2(AcO)_4\cdot 4H_2O]\cdot nH_2O$ where M is Ni(II) and Co(II), n is 1 and 0, respectively, (0.66 g for Ni(II), 0.64 g Co(II), 1.0 mmol) in hot EtOH (15 cm^3) were slowly added with stirring to a solution of N,N′-[3,3′-{ethane-1,2-di-yl-bis(oxy)bis(3,1-phenylene)}bis(N′-hydroxy)-2-(hydroxyimino)acetimidamide], (L^1), (0.21 g, 0.5 mmol) in EtOH (50 cm^3) at 60-65 °C temperature. The pH of the mixtures was *ca.* 4-5. NaOH was added to the mixture to adjust the pH of the mixture and to complete the precipitation after the mixture had been heated on a water-bath 5 h. Finally, the mixtures were centrifuged several times with water and ethanol and the product dried in vacuo to yields a different color powder. These compounds are soluble in DMF and DMSO, and insoluble water and ethanol.

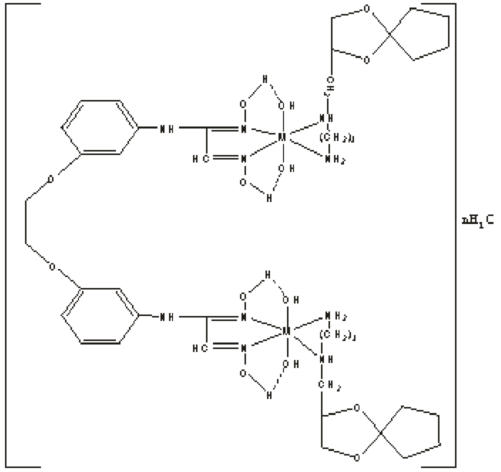


Figure 2. Proposed suggested structure of the octahedral [L1M2L22(OH)4]·nH2O (M: Cu(II), Ni(II), Co(II) and Cd(II))

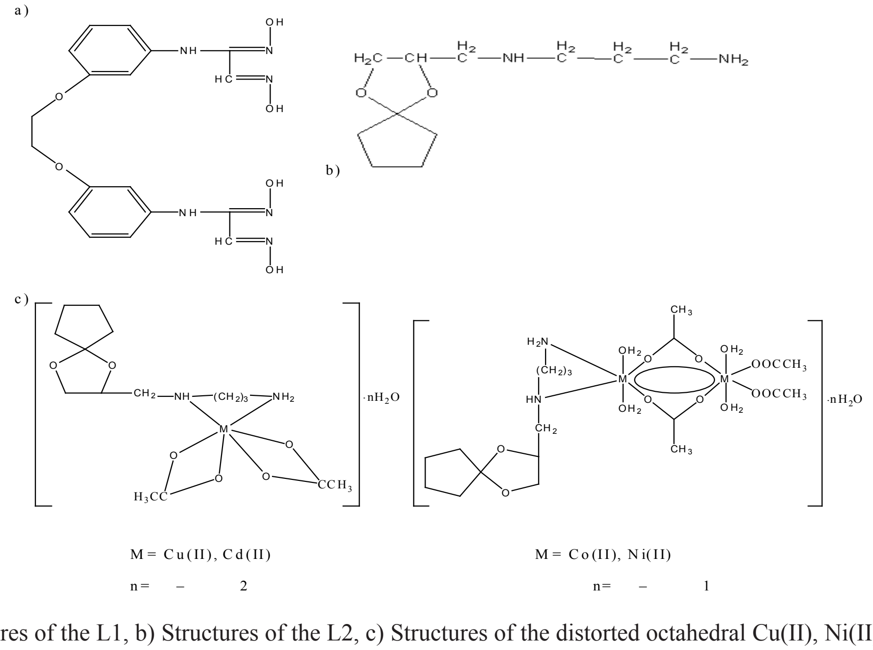


Figure 1 a) Structures of the L1, b) Structures of the L2, c) Structures of the distorted octahedral Cu(II), Ni(II), Co(II) and Cd(II) complex of the (L2)

Table 1. Analytical and Physical Data of the Ligands and Their Complexes

Compounds	Formula (F.W.) g/mole	Color	Yield (%)	μ_{eff}/atom (B.M.)	Λ_M	Elemental analyses Calc. (found), %		
						C	H	N
L^1	$C_{18}H_{20}N_6O_6$ (416.39)	Light Yellow	73.55	—	—	51.92 (51.54)	4.84 (5.08)	20.18 (19.87)
L^2	$C_{11}H_{22}N_2O_2$ (214.0)	Colorless	46.73	—	—	61.68 (61.38)	10.28 (10.42)	13.08 (13.23)
$[L^1Cu_2L^2_2(OH)_4]\cdot H_2O$	$Cu_2C_{40}H_{70}N_{10}O_{15}$ (1058.13)	Light Brown	78.56	1.61	8.27	45.40 (45.56)	6.67 (6.72)	13.24 (13.11)
$[L^1Ni_2L^2_2(OH)_4]\cdot 3H_2O$	$Ni_2C_{40}H_{74}N_{10}O_{17}$ (1084.46)	Brick red	72.27	2.87	12.54	44.30 (44.44)	6.88 (6.74)	12.92 (13.07)
$[L^1Co_2L^2_2(OH)_4]\cdot 2H_2O$	$Co_2C_{40}H_{72}N_{10}O_{16}$ (1066.92)	Dark Red	70.32	3.95	14.75	45.03 (44.82)	6.80 (6.51)	13.13 (13.20)
$[L^1Cd_2L^2_2(OH)_4]\cdot 2H_2O$	$Cd_2C_{40}H_{72}N_{10}O_{16}$ (1173.88)	Yellow	58.74	Dia	15.02	40.93 (40.54)	6.18 (6.02)	11.93 (11.77)

$\Lambda_M = (\Omega^{-1}cm^2mol^{-1})$

RESULTS and DISCUSSION

The ligand used for this work, N,N′-[3,3′-{ethane-1,2-di-yl-bis(oxy)bis(3,1-phenylene)}bis(N′-hydroxy)-2-(hydroxyimino)acetimidamide], (L^1), contains amino and *vic*-dioximes groups. Its synthesis was accomplished in 73.55% yield by the reaction of 1,2-bis(m-aminophenoxy)ethane and *anti*-chloroglyoxime in ethanol. The impurities were checked by N,N′-[3,3′-{ethane-1,2-di-yl-bis(oxy)bis(3,1-phenylene)}bis(N′-hydroxy)-2-(hydroxyimino)acetimidamide], (L^1), gives dinuclear complexes $[L^1Cu_2L^2_2(OH)_4]\cdot H_2O$, $[L^1Ni_2L^2_2(OH)_4]\cdot 3H_2O$, $[L^1Co_2L^2_2(OH)_4]\cdot 2H_2O$ and $[L^1Cd_2L^2_2(OH)_4]\cdot 2H_2O$ with Cu(II), Ni(II), Co(II) and Cd(II) complexes of 6,7-O-cyclopentlydine-1-amino-4-azaheptane (L^2), respectively. The analytical data of these complexes indicate 1:2:2 ligand^1:metal:ligand^2 stoichiometry. Additional analytical data are given in Tables 1-2. Attempt to crystallize the ligand complexes from different solvents were failure. The structure of the L^1 and L^2 ligands were confirmed by a combination of elemental analyses, ^{1}H-NMR, IR and UV-Vis spectral data.

All of the newly synthesized complexes are stable in air. The results of elemental analyses of the ligands and complexes are in agreement with the chemical formulas.

Magnetic susceptibility measurements provided sufficient data to characterize the structures. The copper(II), nickel(II) and cobalt(II) complexes are paramagnetic. Their magnetic susceptibilities are 1.61 B.M. for $[L^1Cu_2L^2_2(OH)_4]\cdot H_2O$, 2.87 B.M. for $[L^1Ni_2L^2_2(OH)_4]\cdot 3H_2O$ and 3.95 B.M. for $[L^1Co_2L^2_2(OH)_4]\cdot 2H_2O$. The alternative chemical environments will give four (O-H···O) bridge protons between hydroxyl ions with –OH groups in the *anti*-form of L^1. The complex of Cd(II) ion, $[L^1Cd_2L^2_2(OH)_4]\cdot 2H_2O$, is diamagnetic. All of the complexes are proposed to be octahedral.

Based on the elemental analyses, spectroscopic characterization and magnetic studies, these binuclear complexes are presumed to have the coordination environment shown in Figure 2.

The infrared spectra of the *vic*-dioxime ligand (L^1), 1,3-dioxolane containing amine ligand (L^2) and their metal complexes have been studied in order to characterize their structures. The relevant IR bands and their assignment are listed in Table 2. Generally, oximes are characterized by three IR absorption bands at 3370-3436 cm^{-1} (O-H$_{str.}$), 1593-1622 cm^{-1} (C=N$_{str}$) and 1078-1055 cm^{-1} (N-O$_{str.}$) [24,25]. In order to study the bridging of the dioxime ligand to the metal in the complexes, the IR spectrum of the free oxime ligand was compared with the spectra of the metal complexes. In the IR spectrum of L^2, the characteristic peaks are at 3361-3203 cm^{-1}, which are assigned to ν(N-H) and ν(-NH$_2$) and 1111 cm^{-1} that is assigned to the ν(C-O-C) group [14,23]. These values are in harmony with previously reported diaminoglyoxime derivatives [26].

In the new complexes of amine and *vic*-dioxime, the oxime C=N stretching vibrations are shifted to 1615-1622 cm^{-1}. These observations indicate the involvement of the nitrogen atom of the azomethine C=N group. In the IR spectra of the binuclear complexes weak O-H···O deformation vibrations are observed at *ca.* 1675-1686 cm^{-1} [12,18]. The disappearance of the OH stretching band and the shift of the C=N band to lower frequency in the IR spectra of binuclear complexes may be attributed to N,N-chelation. N-H stretching vibrations are positioned at near 3350 cm^{-1}. Aromatic C-H stretching vibrations in the L^1 are 3220-3065 cm^{-1}, whereas, aliphatic C-H stretching vibrations in the L^1 and L^2 are 2930-2857 and 2934-2873 cm^{-1}, respectively. At the same time, the infrared bands observed near 3361 and 3203 cm^{-1}, which are assigned to the –NH- and –NH$_2$ frequency is shifted to lower frequency after complexation with respect to the free L^2, whereas, the –NH- and –NH$_2$ frequency is shifted to higher frequency after complexation with respect to the free L^1. The strong absorption at 3356-3337 cm^{-1} in the complexes can be assigned to ν(-NH$_2$) of the intramolecular hydrogen bonded 1,3-diaminopropane moiety [27]. In the spectra of the Cu(II), Ni(II), Co(II) and Cd(II) complexes a few new bands occur at lower regions 461, 467, 455 and 453 cm^{-1}, and 521, 514, 518 and 520 cm^{-1} which are attributed to the ν(M-O) and ν(M-N) vibrations, respectively [28-32].

In order to identify structures of the amino and *vic*-dioxime ligands in solution, the ^{1}H-NMR spectra was recorded in DMSO-d$_6$ or $CDCl_3$.

The ^{1}H-NMR spectrum of *vic*-dioxime ligand (L^1) in DMSO-d$_6$ resulted in peak corresponding to the aromatic protons at 7.32-6.76 ppm as multiplet. In the spectrum of L^1, the N-H protons in the neighbored of oxime groups as multiplet at 6.49-6.11 ppm were exhibited. The singlets at 11.92, 11.45, 10.89 and 10.07 ppm are assigned to the hydroxyl protons of *vic*-dioxime, which disappeared upon addition of D_2O. The singlets for the oxime groups showed that the L^1 was *anti*-configuration [33,34]. The spectrum also show singlets at 4.27-1.18 ppm –CH_2 protons. In the proton NMR spectrum of the ligand, the resonance observed at 8.27 and 7.96 ppm as two singlets were assigned to the azomethine protons of the oxime groups (HC=N-).

In the ^{1}H-NMR spectrum of L^2, there are two characteristic peaks, 1.75 and 2.40-3.20 ppm, which are attributable to the –NH- and –NH_2 groups, which were also identified by D_2O exchange [35] and –CH_2 groups, respectively. There is another –O-CH_2- peaks at 4.03 ppm as multiplet. These are in good agreement with literature.

^{1}H-NMR spectra of the Cu(II), Ni(II) and Co(II) complexes could not be taken because of their paramagnetic character. At this time, since Cd(II) complex could not be soluble from DMSO and DMF, ^{1}H-NMR spectra of the Cd(II) complex could not be taken clearly.

The UV-Vis spectra of the ligands and their complexes were recorded in DMSO-DMF mixture solution in the wavelength range from 200 to 1100 nm. The spectra showed a sharp and intense two bands observed at 260-320 and 370-425 nm for free L^1 [36,37] and 216-290 and 320-400 nm for free L^2 ligand [23,38] are reasonably accounted for $\pi\rightarrow\pi^*$ and $n\rightarrow\pi^*$ transitions.

The electronic spectrum of the $[L^1Cu_2L^2_2(OH)_4]\cdot H_2O$ complex shows a shoulder at 625-750 nm, assignable to $^2B_{1g}\rightarrow{}^2E_g$ transition of the metal ion, suggesting a octahedral geometry [23,39].

The electronic spectrum of the $[L^1Ni_2L^2_2(OH)_4]\cdot 3H_2O$ complex has absorption bands at 435 and 740-775 nm assigned to the $^3A_{2g}\rightarrow{}^3T_{2g}(P)$ and $^3A_{2g}\rightarrow{}^3T_{1g}(F)$ transition, indicating a high-spin octahedral configuration [23,40].

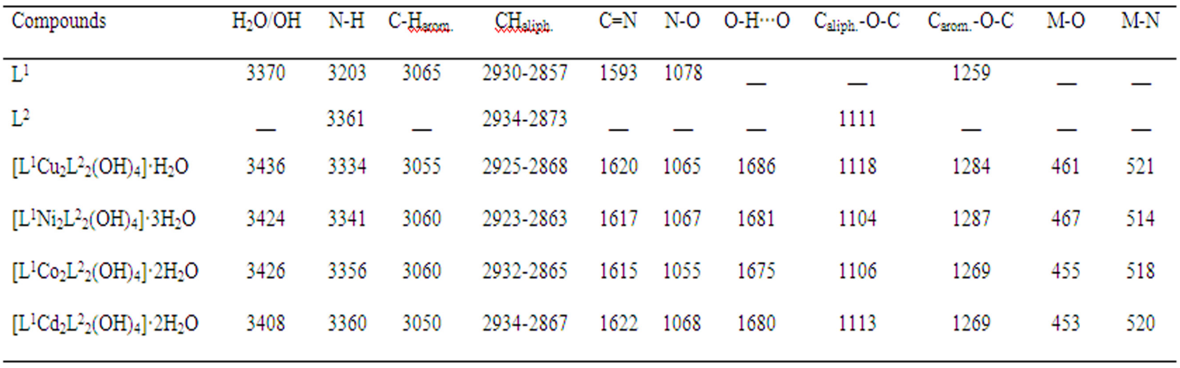
The electronic spectrum of the $[L^1Co_2L^2_2(OH)_4]\cdot 2H_2O$ complex has absorption bands at 515 and 735-790 nm attributable to the $^4T_{1g}(F)\rightarrow{}^4A_{2g}(F)$ and $^4T_{1g}(F)\rightarrow{}^4T_{2g}(F)$ transitions, suggesting a high-spin octahedral geometry around the Co(II) ions [23,41].

A presumably octahedral structure is suggested for the diamagnetic $[L^1Cd_2L^2_2(OH)_4]\cdot 2H_2O$ complexes [37]. This electronic spectra of this complex shows absorption bands at 370 and 430 nm, which are attributed to the charge transfer transitions from the ligand to metal ions and from the metal ions to ligand [42]. The suggested general structure of all the complexes is shown in Figure 2.

The complexes are non-electrolytes as shown by their molar conductivity (Λ_M) measurements in DMSO, which are in the range for Cu(II), Ni(II), Co(II) and Cd(II) complexes at 8.27, 12.54, 14.75 and 15.02 $\Omega^{-1}cm^2mol^{-1}$ [22,43], respectively (Table 1).

The decomposition temperature and the weight losses of the complexes and the ligands were taken from the TGA data. The TGA curves showed that the thermal decomposition of the complexes takes place in three steps. Thermogravimetric studies of all the complexes showed weight loss up to 47.25 ºC, indicating existence of water molecules in the complexes. The inflation of the TGA curves of all the complexes at a temperature below 700.0 ºC, indicates the decomposition of the fully organic part of the chelate, leaving metallic oxide at the final temperature [44].

Table 2. Characteristic IR Bands (cm-1) of the Ligands and Their Complexes in KBr Pellets



Compounds	H_2O/OH	N-H	C-H$_{arom.}$	CH$_{aliph.}$	C=N	N-O	O-H···O	C$_{aliph.}$-O-C	C$_{arom.}$-O-C	M-O	M-N
L^1	3370	3203	3065	2930-2857	1593	1078	_	_	1259	_	_
L^2	_	3361	_	2934-2873	_	_	_	1111	_	_	_
$[L^1Cu_2L^2_2(OH)_4]\cdot H_2O$	3436	3334	3055	2925-2868	1620	1065	1686	1118	1284	461	521
$[L^1Ni_2L^2_2(OH)_4]\cdot 3H_2O$	3424	3341	3060	2923-2863	1617	1067	1681	1104	1287	467	514
$[L^1Co_2L^2_2(OH)_4]\cdot 2H_2O$	3426	3356	3060	2932-2865	1615	1055	1675	1106	1269	455	518
$[L^1Cd_2L^2_2(OH)_4]\cdot 2H_2O$	3408	3360	3050	2934-2867	1622	1068	1680	1113	1269	453	520

CONCLUSION

A novel, vic-dioxime ligand, N,N′-[3,3′-{ethane-1,2-di-yl-bis(oxy)bis(3,1-phenylene)}bis(N′-hydroxy)-2-(hydroxyimino)acetimidamide], L^1, derived from 1,2-bis(m-aminophenoxy)ethane with anti-chloroglyoxime. Then, some transition metal complexes of 6,7-O-Cyclopentlydine-1-amino-4-azaheptane, L^2, have been prepared. These metal complexes of L^2 used as metal salts with vic-dioxime ligand (L^1) have been reacted and new metal complexes of vic-dioxime-amine mixture have been obtained. The analytical data and spectroscopic studies suggest that the complexes were of the general formula $[L^1M_2L^2_2(OH)_4]\cdot nH_2O$ where M is Cu(II), Ni(II), Co(II) and Cd(II), n is 1, 3, 2, 2, respectively. According to the ^{1}H-NMR and IR data of the vic-dioxime ligand, L^1, and amine ligand, L^2, are coordinated with the metal ions through the hydroxyimino nitrogens and amine nitrogens.

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