

**Synthesis and Spectral Studies on
Cobalt(II), Nickel(II), Copper(II),
Palladium(II), Platinum(II, IV), Zinc(II),
Cadmium(II) and Mercury(II) Complexes
of(1, 2-diaminoethane-*N,N'*-bis(2-
butylidene-3 onedioxime)**

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Abstract

The synthesis of [1,2-diaminoethane-*N,N'*-bis(2-butylidene-3-onedioxime)] [H_2L] and its cobalt(II), nickel(II), copper(II), palladium(II), platinum(II, IV), zinc(II), cadmium(II) and mercury(II) complexes is reported. The compounds were characterised by elemental analyses, spectroscopic methods [I.R, UV-Vis, (1H NMR, and EI mass for H_2L)], molar conductivities, magnetic moments. I.R. spectra show that (H_2L) behaves as a neutral or mononegative ligand depending on the nature of the metal ions. The molar conductance of the complexes in (DMSO) is commensurate with their ionic character. On the basis of the above measurements, a square planar geometry is proposed for Ni(II), Pd(II), and Pt(II) complexes, and an octahedral structure with *trans* dichloro ligands at the axial position is proposed for Pt(IV) complex. While [Cu(II) and Co(II)] and [Zn(II), Cd(II) and Hg(II)] adopted a distorted tetrahedral and tetrahedral geometry, respectively.

Introduction:

Oxime compounds and their complexes with transition metals have played a great importance in medicine, industry, chemistry and biochemistry.(1) Compounds such as 2-pyridine aldoxime methiodide

(PAM-2) and diacetylmonoxime (DAM) have been used as antidotes for organo-phosphorus poisoning(2). Organic chelating ligands containing the oxime functional group have been used extensively in analytical chemistry for the detection or separation of metals (3-6). One of the analytical applications of oxime compounds is their uses as organic precipitants for transition metals. Dimethylglyoxime was used for the determination of nickel and palladium metal ions. (7) Moreover, dimethylglyoxime and salicyladoxime have been used as extractant for nickel and copper minerals, respectively .(8) On the other hand, oxime compounds have been used for the analysis of Co(II) in vitamin B₁₂ and Au(III) in rheumath pharmaceutical sample.(9) Oxime complexes of transition metals were used as a powerful catalysis in organic reactions. Hampl and co-workers (10) reported the reactivity of some 2-pyridineketoximes complexes in the cleavage of *p*-nitrophenylesters of carboxylic acid in water. While Yatsimirsky and co-workers(11) showed that Pd(II), Mn(II) and Cd(II) complexes with 2,6-diacetylpyridinedioxime increase the rate constant of the cleavage of 4-nitrophenyl acetate.

Recently, workers (12) reported that palladium(II) complex with di- μ -chloro-*bis*(benzaldehydeoxime) has a robust catalysis for the Heck reaction of aryl halides. Transition metal complexes of *vic*-dioximes are of particular interest as biological model compounds. Numerous chemical studies have been made on the cobalt(II)-cobalt(III)-*bis*(dimethylglyoxime)system which has been called a model system for the B₁₂ moiety (13,14). The antitumor and antimicrobial activities of Co(II) complexes of mono and dimethylglyoxime have been reported. (15,16) In this paper, the preparation and characterisation of Co(II), (Ni(II), Cu(II), Pd(II), Pt(II), Pt(IV), Zn(II), Cd(II) and Hg(II) complexes of [1,2-diaminoethane-*N,N'*-*bis*(2-butyldine-3-onedioxime)] ligand are reported .

Experimental

Reagents were purchased from Fluka & Redial – Dehenge Chemical Co. IR spectra were recorded as (KBr) or (CsI) discs using a Pye-Unicom SP3–300S and a Perkin-Elmer 1720X Series FTIR spectrophotometer. Electronic spectra of the prepared compounds were measured in the region (200 – 1100) nm for 10⁻³ M solutions in (DMSO) at 25°C using a Shimadzu, 160 spectrophotometer with

1.000 \pm 0.001 cm matched quartz cell. Mass spectra for the ligand was obtained by Electron-Impact (EI) on a Shimadzu GCMS QPA 1000 spectrometer. ^1H NMR spectrum for ligand was recorded in (DMSO- d_6) using a Jeol EX270 MHz instrument with a tetramethylsilane (TMS) as an internal standard. The metal contents of the complexes were determined by atomic absorption (AA) technique using a Shimadzu AA 680G atomic absorption spectrophotometer. Elemental microanalyses were performed on a (C.H.N.) analyser, model 1106(Carlo-Erba). Electrical conductivity measurements of the complexes were recorded at 25°C for 10^{-3} M solutions of the samples in dimethylsulfoxide (DMSO) using a PW 9526 digital conductivity meter. Magnetic measurements were recorded on a Bruker BM6 instrument at 298°K following the Faraday's method.

Synthesis of the ligand and its metal complexes

Synthesis of [1,2-diaminoethane-*N,N'*-bis(2-butyldine-3-oxedioxime)] [H₂L]: A solution of ethylenediamine (0.50g, 8 mmole) in methanol (20ml) was added slowly to a mixture of diacetyl monoxime (1.68g, 16.6 mmole) dissolved in methanol (20ml). The reaction mixture was refluxed for two hrs. Then stirred at room temperature for a further 1 hr. . A white solid was collected by filtration, recrystallised from a mixture of hot methanol / H₂O, and dried under vacuum for 24 hrs. to give [H₂L] as a white solid. Yield 2.1 g, (56%), m.p. (164 - 166°C).

Synthesis of 1,2-diaminoethane-*N,N'*-bis (2-butyldine-3-oxoximato)nickel(II) chloride monohydrate [Ni(HL)]Cl.H₂O (1)

A solution of [H₂L] (0.1g, 0.8 mmole), in methanol (20 ml) was added slowly to a stirred solution of nickel(II) chloride hexahydrate (0.2g, 0.8 mmole) in hot methanol (10ml). The resulting mixture was heated under reflux for one hr. during which time the solution became red in colour. The solution was concentrated by evaporating methanol at room temperature. A red solid was formed; this was collected by filtration, washed with dry ethyl ether (10 ml) and dried under vacuum to give 0.16g, (67%) yield of the title compound, m.p. (dec.) (270 - 273°C).

Synthesis of 1, 2-diaminoethane-*N,N'*-bis(2-butyldine-3-oxoximato)palladium(II) chloride monohydrate [Pd(HL)]Cl.H₂O (2)

The method used to prepare the complex [Pd(HL)] Cl.H₂O was similar to that used for [Ni(HL)] Cl H₂O, using palladium(II) chloride (0.1g, 0.5mmole) in place of (NiCl₂.6H₂O). The quantities of other reagents used were adjusted accordingly. An identical work-up

procedure was used to give 0.1g (56%) yield of the title compound as a red solid, m.p. (dec.) (278 - 280°C).

Synthesis of 1,2-diaminoethane-*N,N'*-bis(2-butyldin-3-onedioximato) platinum(II) chloride [Pt(HL)]Cl. (3)

The method used to prepare the complex [Pt(HL)] Cl was analogous to that used for [Ni(HL)]Cl.H₂O, using K₂PtCl₄ (0.1g, 0.2 mmole) in place of (NiCl₂.6H₂O). The quantities of other reagents used were adjusted accordingly. An identical work-up procedure was used to give 0.08g (80%) yield of the title compound as a dark brown solid, m.p. (dec.) (280 - 282°C).

Synthesis of 1,2-diaminoethane-*N,N'*-bis(2-butyldin-3-onedioximato) copper (II) chloride monohydrate [Cu(HL)]Cl.H₂O (4):

The method used to prepare the complex [Cu(HL)]Cl.H₂O was similar to that used for [Ni(HL)]Cl.H₂O, using copper(II) chloride dihydrate (0.19 g, 1.15 mmole) in place of (NiCl₂.6H₂O). The quantities of other reagents used were adjusted accordingly. An identical work-up procedure was used to give 0.32g (86%) yield of the title compound as a green solid, m.p. (dec.) (248 - 250°C).

Synthesis of 1,2-diaminoethane-*N,N'*-bis(2-butyldin-3-onedioximato) cobalt (II) chloridemonohydrate [Co(HL)]Cl.H₂O (5):

The method used to prepare the complex [Co(HL)]Cl.H₂O was analogous to that used for [Ni(HL)]Cl.H₂O, using cobalt(II) chloride hexahydrate (0.2g, 0.84 mmole) in place of (NiCl₂.6H₂O). The quantities of other reagents used were adjusted accordingly. An identical work-up procedure was used to give 0.21g (81%) yield of the title compound as a pale-green solid, m.p. (dec.) (250 - 252°C).

Synthesis of *trans*-dichloro-1,2-diaminoethane-*N,N'*-bis(2-butyldin-3-one dioximato) platinum(IV) chloride [Pt(HL) Cl₂]Cl (6):

The method used to prepare the complex [Pt(HL)Cl₂] Cl was similar to that employed to prepare the complex [Ni(HL)]Cl.H₂O, using K₂PtCl₆ (0.21g, 0.44 mmole) in place of (NiCl₂.6H₂O). The quantities of the other reagents used were adjusted accordingly. An identical work-up procedure was used to give 0.15g (65%) yield of the title compound as a dark red-brown solid, m.p. (dec.) (295 - 297°C).

Synthesis of 1,2-diaminoethane-*N,N'*-bis(2-butyldin-3-onedioxime)zinc(II) dichloride monohydrate [Zn(H₂L)] Cl₂.H₂O (7):

The method used to prepare the complex [Zn(H₂L)]Cl₂.H₂O was similar to that used for [Ni(HL)]Cl.H₂O, using zinc(II) chloride (0.06g, 0.44 mmole) in place of (NiCl₂.6H₂O). The quantities of other reagents used were adjusted accordingly. An identical work-up

procedure was used to give 0.1g (71%) yield of the title compound as a white solid, m.p. (dec.) (230 - 232°C).

Synthesis of 1,2-diaminoethane-*N,N'*-bis(2-butyldin-3-oxedioxime) cadmium(II) dichloride monohydrate [Cd(H₂L)]Cl₂.H₂O (8): The method used to prepare the complex [Cd(H₂L)]Cl₂.H₂O was analogous to that for [Ni(HL)]Cl₂.H₂O, using cadmium(II) chloride dihydrate (0.09g, 0.44 mmole) in place of (NiCl₂.6H₂O). The quantities of other reagents used were adjusted accordingly. An identical work-up procedure was used to give 0.12g (75%) yield of the title compound as a white solid, m.p. (dec.) (273 - 275°C).

Synthesis of 1,2-diaminoethane-*N,N'*-bis(2-butyldin-3-oxedioxime) mercury (II) dichloride monohydrate [Hg(H₂L)]Cl₂.H₂O (9): The method used to prepare the complex [Hg(H₂L)]Cl₂.H₂O was similar to that employed to prepare complex [Ni(HL)]Cl₂.H₂O, using mercury(II) chloride (0.1g, 0.4 mmole) in place of (NiCl₂.6H₂O). The quantities of other reagents used were adjusted accordingly. An identical work-up procedure was used to give 0.11g (61%) yield of the title compound as a white solid, m.p. (dec.) (275 - 277°C).

Results and discussion

Synthesis of the ligand: Since EI-Tabl and Kashar (17), failed to prepare the free imine oxime ligand, derived from the condensation of diacetyloxime and 1,2-diaminopropane. In the present work, however, the new ligand was successfully prepared according to the general method shown in Scheme (1). The (I.R) spectrum for [H₂L] Fig. (1a) displayed two bands at (1609) and (1473) cm⁻¹ due to ν (C=N) stretching for the imine and oxime groups respectively (18). The broad band at (3145) cm⁻¹ is attributed to the ν (O-H) stretching of the oxime group. The strong bands at (1000) and (918) cm⁻¹ are attributed to ν (N-O) stretching. The (U.V.-Visi) spectrum Fig. (2a) exhibits a high intense absorption peak at (293 nm) (34130cm⁻¹) (ϵ_{\max} =1978 molar⁻¹cm⁻¹) which is assigned to overlap of ($\pi \rightarrow \pi^*$) and ($n \rightarrow \pi^*$) transitions (19). The EI (+) mass spectrum of the ligand (Fig.(3), shows the parent ion peak at (m/z = 226), which corresponds to (M)⁺, and the fragments at, 209= [M-(OH)]⁺, 192 = [M-{(OH)₂}]⁺, 166 = M-{(OH)₂-CN}]⁺, 151 = [M-{(OH)₂-CN-CH₃}]⁺, 126 = [M-{(OH)₂-CN-CH₃-CN}]⁺, 113 = [M-{(OH)₂-CN-CH₃-CN-CH}]⁺, 97 = [M-{(OH)₂-CN-CH₃-CN-CH-NH₂}]⁺, 69= [M-{(OH)₂-CN-CH₃-CN-CH-NH₂-CNH₂}]⁺, 56 = [M-{(OH)₂-CN-CH₃-CN-CH-NH₂-CNH₂-CH}]⁺.

The $^1\text{H NMR}$ spectrum of $[\text{H}_2\text{L}]$, Fig.(4) in (DMSO-d^6) shows protons (OH) of the oxime appear as a broad signal at (11.46) ppm. This resonance was disappeared upon addition of (D_2O) to the solution.(20) The CH_2 protons are equivalent and appear as a singlet at (3.70) ppm. As can be seen from the spectrum, the two sharp singlets at (2.02) and (1.89) ppm are due to the methyl groups adjacent to carbon (C-1) and carbon (C-2), respectively. However each, peak equivalent to six protons (21, 22),

Synthesis of the complexes: The reaction of $[\text{H}_2\text{L}]$ with $[\text{M}^{n+}]$ metal ions (where: $n= 2$, $\text{M}= \text{Ni, Pd, Pt, Co, Cu, Zn, Cd}$ and Hg ; $n= 4$, $\text{M}= \text{Pt}$), was carried out in EtOH under reflux. These complexes are stable in solution and electrolytes, Table (3). The analytical and physical data Table (1) and spectral data Tables (2) and (3) are compatible with the suggested structures Fig.(5).

(IR) Spectra: The (I.R) spectral data of the complexes are presented in (Table 2). The strong $\nu(\text{C}=\text{N})$ stretching bands in the free ligand at (1609) and (1473) cm^{-1} for the imine and oxime groups respectively are shifted, and appear at (1596, 1450), (1590, 1465), (1576, 1462) and (1540, 1460), (1573, 1454), (1564, 1440), (1590, 1460), (1605, 1427) and (1598, 1461) cm^{-1} for the compounds $[\text{Ni}(\text{HL})]\text{Cl}\cdot\text{H}_2\text{O}$ (Fig. 1b), $[\text{Pd}(\text{HL})]\text{Cl}\cdot\text{H}_2\text{O}$ (Fig. 1c), $[\text{Pt}(\text{HL})]\text{Cl}\cdot\text{H}_2\text{O}$, $[\text{Pt}(\text{HL})\text{Cl}_2]\text{Cl}$ (Fig. 1d), $[\text{Zn}(\text{H}_2\text{L})]\text{Cl}_2\cdot\text{H}_2\text{O}$, $[\text{Cd}(\text{H}_2\text{L})]\text{Cl}_2\cdot\text{H}_2\text{O}$, $[\text{Hg}(\text{H}_2\text{L})]\text{Cl}_2\cdot\text{H}_2\text{O}$, $[\text{Cu}(\text{HL})]\text{Cl}\cdot\text{H}_2\text{O}$ and $[\text{Co}(\text{HL})]\text{Cl}\cdot\text{H}_2\text{O}$, respectively. These bands were assigned to the $\nu(\text{C}=\text{N})$ stretches of reduced bond order. This can be attributed to the delocalisation of metal electron density into the ligand π -system (23). The strong $\nu(\text{N}-\text{O})$ stretching bands at (918) and (1000) cm^{-1} for the free ligand are shifted markedly to higher frequencies by *ca.*(211) cm^{-1} . This is presumably due to the complexation with the metal ions (24). For these complexes, the two N-O bonds are probably unequal. These results are in a good agreement with those reported by Bigatto and co-workers.(25) For the zinc group complexes, the $\nu(\text{O}-\text{H})$ stretching band of the oxime group in the free ligand at (3145) cm^{-1} is still present in the region (3121–3235) cm^{-1} (23). For other complexes, the weak and broad bands appeared in the region (2580 – 2405) cm^{-1} and (1765 – 1736) cm^{-1} are due to the $\nu(\text{O}\dots\text{H}-\text{O})$ stretching and $\delta(\text{O}\dots\text{H}-\text{O})$ bending for the hydrogen bond respectively, in which the ligand loses a proton and a hydrogen bonding will be formed. However, these bands are absent in the free ligand.(26) The weak bands at *ca.* (550-425), cm^{-1} range were assigned to $\nu(\text{M}-\text{N})$ stretches, indicating that the imine and oxime

nitrogens were involved in coordination with metal ion. The broad band observed in the region (3411-3328) cm^{-1} is due to the $\nu(\text{O-H})$ stretching of lattice water^(17, 27).

Electronic Spectra: The (U.V-Vis) spectra of the complexes [Ni(HL)] Cl.H₂O (1) Fig. (2b), [Pd(HL)] Cl.H₂O (2), [Pt(HL)] Cl.H₂O (3), [Cu(HL)] Cl.H₂O (4) Fig. (2c), [Co(HL)] Cl.H₂O (5) Fig. (2d) and [Pt(HL)Cl₂] Cl (6) displayed two intense peaks in the (U.V.) region at (273-320 nm) range assigned to ligand field and charge transfer transitions. Complex (1) showed another two peaks at (380 nm) and (555 nm), these peaks are assigned to (¹A_{1g} → ¹B_{1g}) (a_{1g} → b_{1g}) and (¹A_{1g} → ¹A_{2g}) (b_{2g} → b_{1g}) (d-d) transitions in a square planar structure (29). However, the first peak at (380nm) was shifted to lower wavelength due to the intensity stealing. This was in a good agreement with the results reported by Aly and co-workers (30) of nickel(II) complexes with imine-oxime ligands. The spectra of complexes (2) and (3) exhibited weak peaks in the visible region at (407 nm) and (418nm), respectively. These bands were assigned to (¹A_{1g} → ¹B_{1g}) (d – d) transition, suggesting a square planar structure around palladium(II) and platinum(II) ion.(29) This is in agreement with the results reported by Martin and co-workers(31) and Alti and co-workers(32) of palladium(II) and platinum(II) complexes with diphenylglyoxime and dimethylglyoxime. The spectrum of complex (4) exhibited a weak broad peak in the visible region at (773 nm) assigned to (²B₂ → ²E) (d-d) transition confirming a distorted tetrahedral structure around copper(II) ion. The broadening of this band is due to the Jahn-Teller effect. In the [Co(HL¹)]Cl.H₂O (5) spectrum the peak at (550 nm) which can be assigned to (⁴A_{2(F)} → ⁴T_{1(P)}) (d-d) transition and suggesting a distorted tetrahedral structure around the cobalt(II) ion.⁽¹⁰²⁾ This is in accordance with the results reported by Aly *et al* for dimethylglyoximato cobalt(II) complexes.(30) Pt(IV) low spin complexes have the electron configuration t_{2g}^6 transforms as ¹A_{1g}. Two-principle spin allowed absorption bands are to be expecting corresponding to transitions from the ¹A_{1g} ground state to the ¹T_{1g} and ¹T_{2g} excited states. Thus complex (6) exhibits two weak peaks in the visible region at (440 nm) and (600 nm), which assigned to (¹A_{1g} → ¹T_{2g}) and (¹A_{1g} → ¹T_{1g}) (d-d) transitions respectively confirming a *trans* octahedral structure around Pt(IV) ion (28). The (U.V.-Vis) spectra of the complexes [Zn(H₂L)]Cl₂.H₂O (7), [Cd(H₂L)]Cl₂.H₂O (8) and [Hg(H₂L)]Cl₂.H₂O (9), respectively showed an intense peak in the (U.V) region at (260-

314 nm). Since the metal ion of the compounds belong to d^{10} system, these peaks were assigned to charge transfer transitions (29).

Magnetic moments: The magnetic moments for the complexes are shown in table (3). The copper(II) (2.18 B. M) and cobalt(II) (4.55 B.M.) complexes have a distorted tetrahedral geometry, however the nickel(II), palladium(II), platinum(II) complexes lie in (0.2-0.44 B.M.) indicating a square planar geometry.

Conductivity measurements: The molar conductance of the complexes in (DMSO), Table (3) lie in the (67-45) $S\ cm^2\ mole^{-1}$ range, indicating their electrolytic nature with (1:1) ratio, except for the zinc group complexes (1), (2), and (3) in which their molar conductance lie in the (158-155) $S\ cm^2\ molar^{-1}$ range, indicating their ionic behaviour with (1:2) ratio (33).

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Table(1) Analytical and physical data of the ligand and its complexes

Empirical Formula	M.wt.	Yield %	M.P. (°C)	Colour	Micro analysis Found, (calc.) %			
					C	H	N	Metal
C ₁₀ H ₁₈ N ₄ O ₂	226.27	56	164 –166	white	(53.07)	(8.01)	(24.76)	-
					52.83	8.20	24.42	-
NiC ₁₀ H ₁₉ N ₄ O ₃ Cl	337.41	67	270-272	red	(35.59)	(5.63)	(16.60)	(17.39)
			(dec.)		35.72	5.76	16.40	17.12
PdC ₁₀ H ₁₉ N ₄ O ₃ Cl	385.42	56	278-280	red	(31.16)	(4.92)	(14.54)	(27.68)
			(dec.)		31.34	4.76	14.36	27.80
PtC ₁₀ H ₁₉ N ₄ O ₃ Cl	473.95	80	280-282	brown	(25.34)	(4.00)	(11.82)	(41.19)
			(dec.)		25.16	4.25	11.74	41.32
PtC ₁₀ H ₁₇ N ₄ O ₂ Cl ₃	526.71	65	295-297	red- brown	(22.80)	(3.23)	(10.64)	(37.06)
			(dec.)		23.08	3.48	10.53	37.24
CuC ₁₀ H ₁₉ N ₄ O ₃ Cl	342.29	86	248-250	green	(35.08)	(5.55)	(16.37)	(18.57)
			(dec.)		35.25	5.66	16.57	18.32
CoC ₁₀ H ₁₉ N ₄ O ₃ Cl	337.66	81	250-252	Pale- green	(35.56)	(5.62)	(16.59)	(17.45)
			(dec.)		35.23	5.80	16.80	17.66
ZnC ₁₀ H ₂₀ N ₄ O ₃ Cl ₂	381.05	71	230-232	white	(31.51)	(5.24)	(14.70)	(17.28)
			(dec.)		31.26	5.40	14.63	16.96
CdC ₁₀ H ₂₀ N ₄ O ₃ Cl ₂	427.58	75	273-275	white	(28.08)	(4.67)	(13.10)	(26.28)
			(dec.)		27.85	4.86	13.35	26.50
HgC ₁₀ H ₂₀ N ₄ O ₃ Cl ₂	515.78	61	275-277	white	(23.28)	(3.87)	(10.86)	(38.89)
			(dec.)		22.96	3.52	10.63	38.61

(Calc.): Calculated (dec.) : Decompos

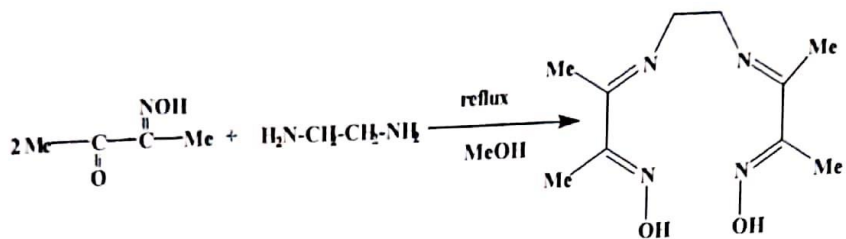
Table (2) I.R Spectral data of the ligand and its complexes(cm^{-1})

Compound	$\nu(\text{O-H})$ oxime	(O-H) H_2O	$\nu(\text{O}\cdots\text{H-O})$	$\delta(\text{O}\cdots\text{H-O})$	$\nu(\text{C=N})$ imine $\nu(\text{C=N})$ oxime	$\nu(\text{N-O})$	$\nu(\text{M-N})$
$[\text{H}_2\text{L}]$	3145 (m)		-	-	1609 (s) 1473 (s)	918 (w) 1001 (s)	-
$[\text{Zn}(\text{H}_2\text{L})]\text{Cl}_2 \cdot \text{H}_2\text{O}$	3235 (m) 3289 (m)	3450 (br.)	-	-	1573 (m) 1454 (w)	1009 (m) 1137 (m)	477 (w)
$[\text{Cd}(\text{H}_2\text{L})]\text{Cl}_2 \cdot \text{H}_2\text{O}$	3209 (s) 3284 (s)	3450 (br.)	-	-	1564 (m) 1440 (m)	1066 (m) 1160 (m)	452 (w)
$[\text{Hg}(\text{H}_2\text{L})]\text{Cl}_2 \cdot \text{H}_2\text{O}$	3121 (s) 3194 (s)	3307 (br.)	-	-	1590 (m) 1460 (w)	1005 (m) 1167 (m)	526 (w) 359 (w)
$[\text{Ni}(\text{HL})]\text{Cl} \cdot \text{H}_2\text{O}$	-	3328 (br.)	2410 (w.br.)	1736 (w br.)	1596 (m) 1450 (m)	1039 (m) 1245 (m)	525 (w) 444 (w)
$[\text{Pd}(\text{HL})]\text{Cl} \cdot \text{H}_2\text{O}$	-	3411 (br.)	2450 (w.br.)	1765 (w.br.)	1590 (m) 1465 (m)	1032 (m) 1251 (m)	522 (w)
$[\text{Pt}(\text{HL})]\text{Cl} \cdot \text{H}_2\text{O}$	-	3336 (m)	2405 (w.br.)	1745 (w br.)	1576 (m) 1462 (m)	1051 (w)	518 (w) 425 (w)
$[\text{Pt}(\text{HL})\text{Cl}_2]\text{Cl}$	-	-	2580-2480 (w.br.)	1745 (w.br.)	1540 (m) 1460 (w)	1090 (m) 1250 (m)	510 (w)
$[\text{Cu}(\text{HL}^1)]\text{Cl} \cdot \text{H}_2\text{O}$	-	3378 (m.br.)	2430 (w.br.)	1756 (w.br.)	1605 (w) 1427 (s)	1077 (s) 1232 (w)	588 (w) 460 (w)
$[\text{Co}(\text{HL}^1)]\text{Cl} \cdot \text{H}_2\text{O}$	-	3441 (br.)	2430 (w.br.)	1762 (w.br.)	1598 (m) 1461 (m)	1007 (m) 1269 (m)	515 (w) 430 (w)

s: strong, vs: very strong, m: medium, w: weak, s,sh: strong sharp, m,sh: medium sharp
br.: broad, o.o.p: out of plane, aliph: aliphatic, arom.: aromatic, ν : stretching, δ :bending

Table (3) Electronic spectral data ⁿ, magnetic moments and conductivity measurement of [H₂L] and its metal complexes

Compound	λ nm	ν cm ⁻¹	ϵ_{max} (molar ⁻¹ , cm ⁻¹)	μ_{eff} (BM)	$\Lambda_M(\Omega^{-1}$ cm ² mol ⁻¹)	Ratio
[H ₂ L]	293	34129.7	1978			
[Zn(H ₂ L)]Cl ₂ .H ₂ O	269	37175	1903		157	(2:1)
[Cd(H ₂ L)]Cl ₂ .H ₂ O	314	31847	1102		158	(2:1)
[Hg(H ₂ L)]Cl ₂ .H ₂ O	264	37879	1750		155	(2:1)
[Ni(HL)]Cl.H ₂ O	273	36630	1667	0.63	53.3	(1:1)
	328	30488	1003			
	380	26316	800			
	555	18018	239			
[Pd(HL)]Cl.H ₂ O	271	36900	1940	0.62	58.46	(1:1)
	301	33222	1431			
	407	24570	611			
[Pt(HL)]Cl.H ₂ O	274	36496	1399	0.30	50.28	(1:1)
	304	32895	1376			
	418	23923	153			
[Pt(HL)Cl ₂]Cl	274	36496	1464	0.33	57.78	(1:1)
	440	22727	166			
	600	16667	66			
[Cu(HL)]Cl.H ₂ O	304	32895	1217	2.14	66.80	(1:1)
	364	27472	1130			
	773	12937	191			
[Co(HL)]Cl.H ₂ O	304	32895	1668	4.1	44.56	(1:1)
	361	27700	1029			
	550	18182	300			



Scheme (1) The synthesis route of the ligand

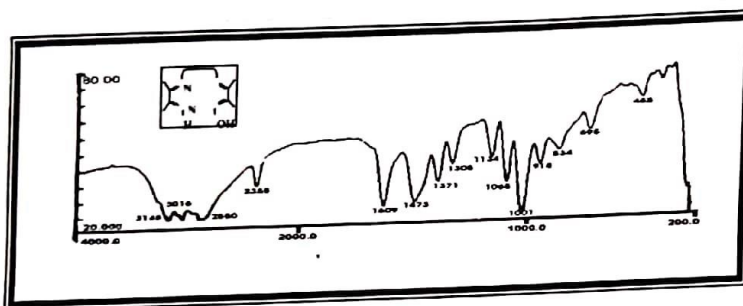


Figure (1a): Infrared spectrum of [H₂L]

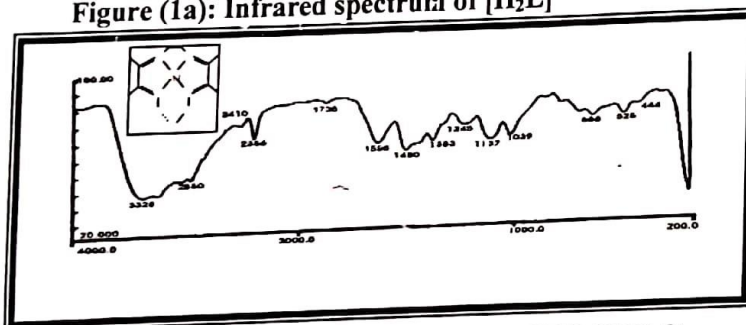


Figure (1b): Infrared spectrum of [Ni(HL)] Cl.H₂O

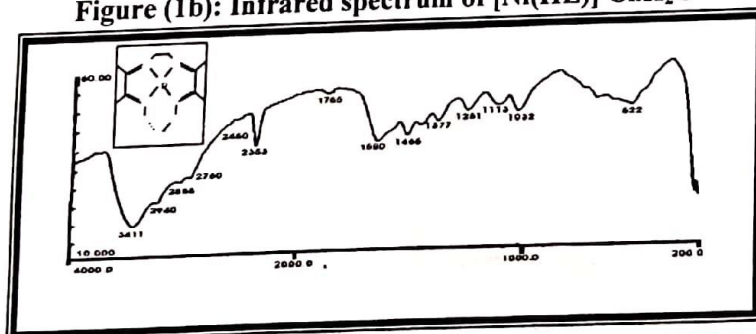


Figure (1c): Infrared spectrum of [Pd(HL)] Cl.H₂O

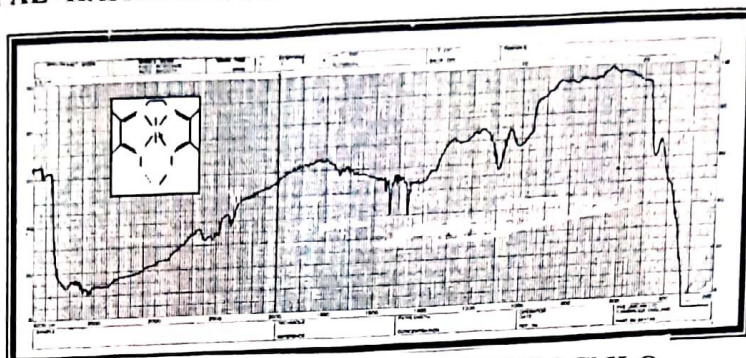


Figure (1d): Infrared spectrum of $[Pt(HL)Cl_2] Cl.H_2O$

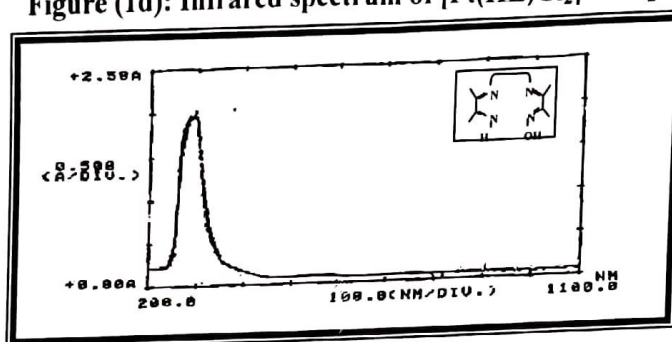


Figure (2a): Electronic spectrum of $[H_2L]$

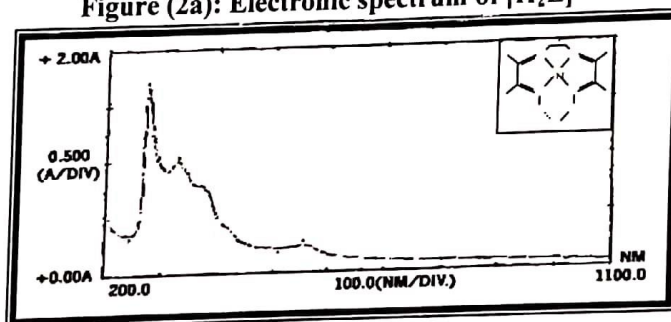


Figure (2b): Electronic spectrum of $[Ni(HL)] Cl.H_2O$

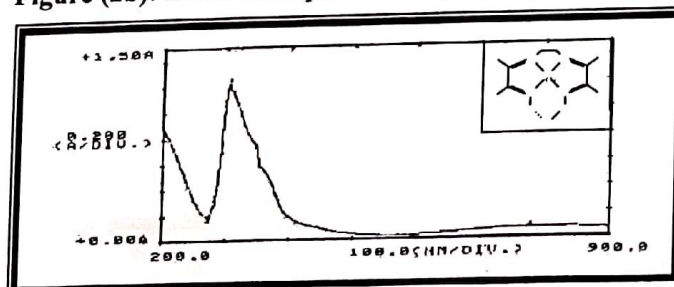


Figure (2c): Electronic spectrum of $[Cu(HL)] Cl.H_2O$

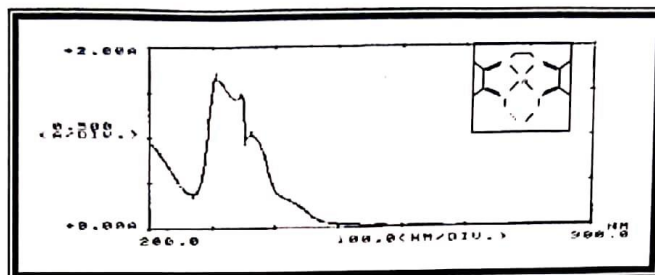


Figure (2d): Electronic spectrum of [Co(HL)] Cl.H₂O

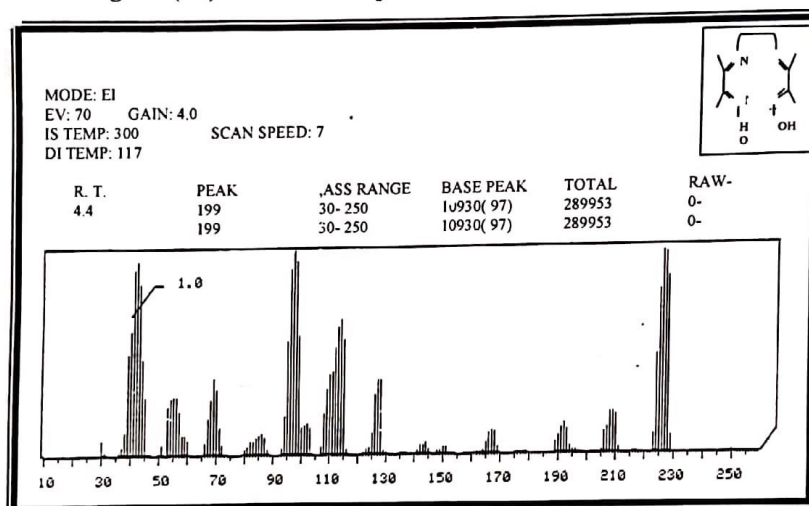


Figure (3) EI mass spectrum of [H₂L]

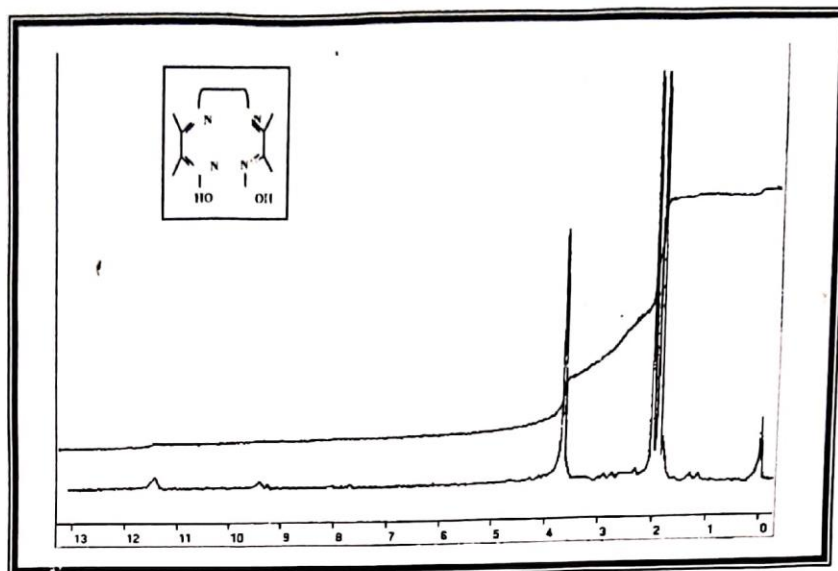


Figure (4) ^1H NMR spectrum of $[\text{H}_2\text{L}]$

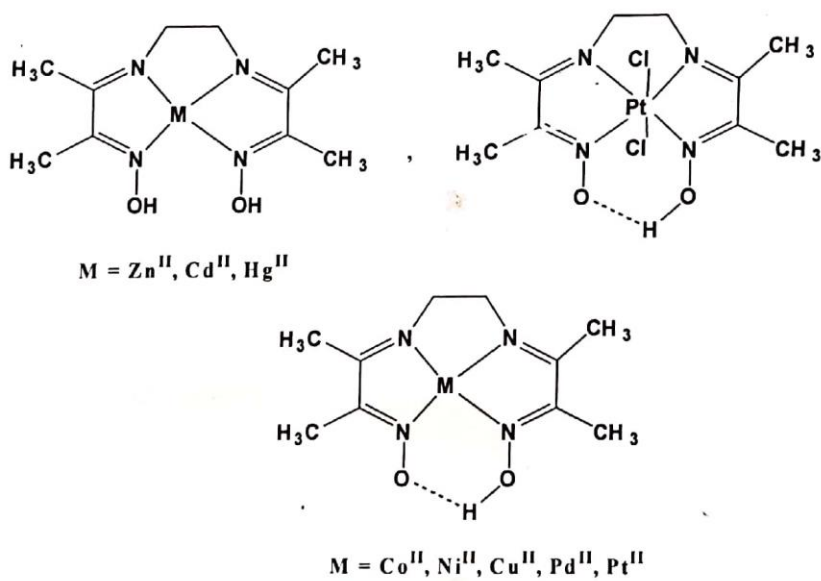


Figure (5) The proposed structures for the complexes

تحضير مع دراسات طيفية لمعقدات
 $(\text{Co}^{\text{II}}, \text{Ni}^{\text{II}}, \text{Cu}^{\text{II}}, \text{Pd}^{\text{II}}, \text{Pt}^{\text{II,IV}}, \text{Zn}^{\text{II}}, \text{Cd}^{\text{II}} \text{ and } \text{Hg}^{\text{II}})$
 مع اليكاند
 (1, 2-diaminoethane-*N,N'*-bis(2-butyldine-3
 onedioxime)

محمد جابر الجبوري وعبدالسلام عبدالكريم عبدالرحمن وصبحي عطية
 قسم الكيمياء ، كلية التربية ابن الهيثم ، جامعة بغداد

الخلاصة

تضمن البحث تحضير اليكاند رباعي السن نوع N_4
 (1, 2-diaminoethane-*N,N'*-bis(2-butyldine-3 onedioxime) (H_2L)
 ومعقداته مع $(\text{Co}^{\text{II}}, \text{Ni}^{\text{II}}, \text{Cu}^{\text{II}}, \text{Pd}^{\text{II}}, \text{Pt}^{\text{II,IV}}, \text{Zn}^{\text{II}}, \text{Cd}^{\text{II}} \text{ and } \text{Hg}^{\text{II}})$
 شخصت جميع المركبات المحضرة بالطرائق الطيفية الآتية [الأشعة تحت الحمراء و
 الأشعة فوق البنفسجية- المرئية و مطيافية التذرية و (طيف الكتلة بتقنية القصف
 الالكتروني $^1\text{HNMR}$, EI) لليكاند] ، كذلك شخصت المركبات بوساطة، التحليل
 الكمي الدقيق للعناصر والحساسية المغناطيسية مع التوصيلة المولارية الكهربائية .
 بينت دراسات طيف الأشعة تحت الحمراء ان اليكاند يسلك سلوكا متعادلا" او سالب الشحنة
 (1-) معتمد على طبيعة الايون الفلزي.
 كذلك بينت دراسات التوصيلية الكهربائية للمعقدات في ال (DMSO) طبيعتها الايونية.