

## Preparation and Spectroscopic Studies of Some Metal Ion Complexes of 2-((4-Formyl-3-Hydroxynaphthalen-2-yl) Diazenyl) Benzoic Acid

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### Abstract

New azo ligand 2-((4-formyl-3-hydroxynaphthalen-2-yl) diazenyl) benzoic acid (H<sub>2</sub>L) was synthesized from the reaction of 2-aminobenzoic acid and 2-hydroxy-1-naphthaldehyde. Monomeric complexes of this ligand, of general formulae [M<sup>II</sup>(L)(H<sub>2</sub>O)] with (M<sup>II</sup> = Mn, Co, Ni, Cu, Zn, Pd, Cd and Hg ) were reported. The compounds were isolated and characterized in solid state by using <sup>1</sup>H-NMR, FT-IR, UV-Vis and mass spectral studies, elemental microanalysis, metal content, magnetic moment measurements, molar conductance and chloride containing. These studies revealed tetrahedral geometries for all complexes except Pd<sup>II</sup> complex is Square planar. The study of complexes formation via molar ratio of (M:L) as (1:1). Theoretical treatments of compounds in gas phase were studied using Hyper Chem-8 program has.

**Key words:** Azo dye, 2-Hydroxy-1-Naphthaldehyde and Azo Complexes

### Introduction:

Azo dyes comprise the largest group of organic reagent used in spectrophotometric analysis. They are found in a variety of industrial applications because of their color fastness. These dyes are characterized by chromophoric azo group (-N=N-) offering a wide range spectrum of colors [1]. They are usually strongly coloured compounds, which can be intensely yellow, red, orange, blue or even green, depending on the exact structure of the molecule. Because of their colour, azo

compounds are tremendous importance as dyes and as pigments for a long time [2, 3]. They also used for coloring consumer goods such as leather, clothes, food, toys, plastic and cosmetics[1]. Infact, about half of the dyes in industrial use today are azo dyes, which are mostly prepared from diazonium salts [4, 5].The azo group possesses excellent donor properties and is important in coordination chemistry [6]. Azo compounds are very important class of chemical compounds receiving

attention in scientific research [7]. The aim of this paper is to synthesize, characterize and study the biological activities of the new tridentate azo ligand [2-((4-formyl-3-hydroxynaphthalen-2-yl) diazenyl) benzoic acid] H<sub>2</sub>L and its metal complexes.

### Materials and Methods:

All reagents were commercially available and used without further purification. Elemental microanalyses (C.H.N.) were performed by using a flash E A 1112 Series elemental microanalyses. IR spectra were obtained by using Perkin – Elmer FT-IR spectrometer. The electronic spectra were carried out by using a Cary 50 Conc. UV-Visible spectra were recorded using spectrophotometer. Thermal analysis studies of the compounds were performed on Perkin – Elmer pyris Diamond DTA / TG.. Chloride was determined by using a 686-Titro processor – 665 Dosimat Metrohm Swiss. Conductivity measurements were made with using a Jenway 4071 digital. Magnetic moments were measured with a magnetic susceptibility balance (Jonson Matthey Catalytic system Division). Mass spectra were obtained by LC-Mass 100 P Shimadzu. NMR spectra (<sup>1</sup>H-NMR) were acquired in DMSO-d<sub>6</sub> solution by using Bruker AMX 400 MHz spectrometer. Metals were determined by using a Shimadzu (A. A) 680 G. Melting points were obtained on a Buchi SMP -20 capillary.

#### Preparation of Ligand: 2-((4-Formyl-3-Hydroxynaphthalen-2-yl) Diazenyl) Benzoic Acid (H<sub>2</sub>L)

(1g, 0.007mol) of 2-aminobenzoic acid was dissolved in a mixture of (2 ml) hydrochloric acid, (20 ml) ethanolic solution, and diazotized at (0 to -5) °C with NaNO<sub>2</sub> solution. The diazonium salt solution was added drop wise with stirring to a cooled ethanolic solution of (1.255g, 0.007mole) of 2-

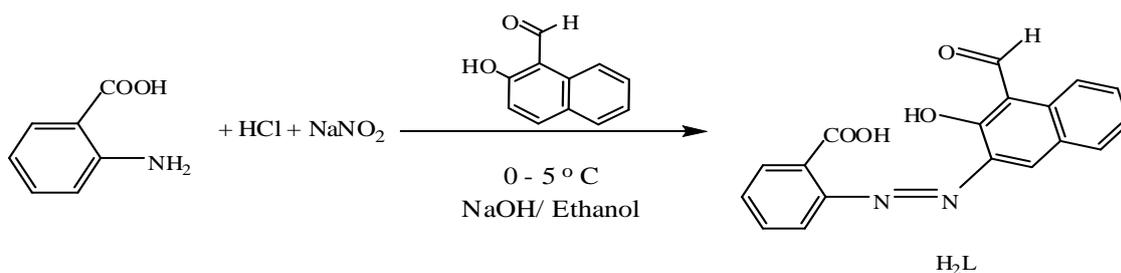
hydroxy-1-naphthaldehyde. (25 ml) of (1 M) NaOH solution was added to the light yellow colored mixture. The precipitate was filtered off and washed several times with (1:1) ethanol: water, mixture then left to dry. After that, the product precipitated yield (84.88%), m. p. (88-90°C). <sup>1</sup>H-NMR (DMSO-d<sub>6</sub>, ppm): 13.2 (s, O-H carboxylic), 9.123(1H, s, O=CH) aldehyde, 7.309-8.515(5H, m, naphthalene-H) arom. 4.49 (s, O-H phenol).

#### Preparation of Complexes:

The preparation of all complexes is essentially the same and so a generic description will be presented. To a solution of (H<sub>2</sub>L) (0.006 mole) in ethanol was added slowly to a solution of metal salt (MnCl<sub>2</sub>.4H<sub>2</sub>O, CoCl<sub>2</sub>.6H<sub>2</sub>O, NiCl<sub>2</sub>.6H<sub>2</sub>O, CuCl<sub>2</sub>, ZnCl<sub>2</sub>, PdCl<sub>2</sub>, CdCl<sub>2</sub> and HgCl<sub>2</sub>) in (ethanol and water) ratio (1:1) with stirring the mixture was refluxed for (5hrs). Elemental micro analysis data, color and yield for the complexes are given in Table (1).

### Results and Discussion:

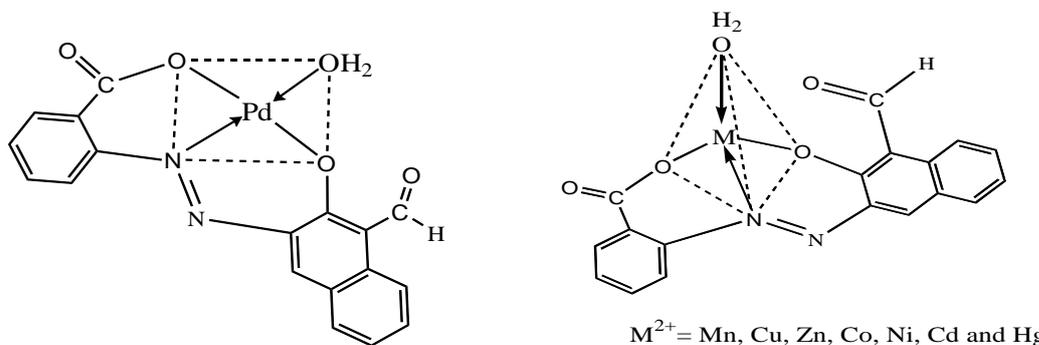
The new (ONO) dentate ligand H<sub>2</sub>L was obtained in good yield by the reaction of 2-aminobenzoic acid and 2-hydroxy-1-naphthaldehyde, Scheme (1). In general the ligand was characterized by elemental microanalysis and physical properties Table (1), FT-IR Table (2), UV-Vis Table (3), mass spectroscopy. Monomeric complexes of the ligand with metal ions were synthesized by heating (1mmol) of each ligand with (1 mmol) of metal salt, using ethanolic. However, in ethanolic, deprotonation of the ligand occur facilitating the formation of the complexes [M<sup>II</sup>(L)(H<sub>2</sub>O)] (M<sup>II</sup> = Mn, Co, Ni, Cu, Zn, Pd, Cd and Hg) are reported, Scheme (2). The analytical data Table (1) agree well with the suggested formula



Scheme 1: Synthesis of the Ligand

Table (1): Analytical and Physical Data of the Ligand and its Complexes

Compounds	Formula M. wt	Color	M. P. °C	Yield %	Elemental micro analysis, found (Calc.)%			
					C	H	N	M
H <sub>2</sub> L	C <sub>18</sub> H <sub>12</sub> N <sub>2</sub> O <sub>4</sub> 320.08	Light yellow	88-90	85	67.16 (67.50)	3.42 (3.78)	8.77 (8.75)	-
[MnHL(H <sub>2</sub> O)]	C <sub>18</sub> H <sub>12</sub> MnN <sub>2</sub> O <sub>5</sub> 391.24	Brown	210d	88	56.02 (55.26)	3.32 (3.09)	7.41 (7.16)	14.41 (14.04)
[CoHL(H <sub>2</sub> O)]	C <sub>18</sub> H <sub>12</sub> CoN <sub>2</sub> O <sub>5</sub> 395.23	Violate	158 d	74	55.11 (54.70)	3.31 (3.06)	7.00 (7.09)	14.43 (14.91)
[NiHL(H <sub>2</sub> O)]	C <sub>18</sub> H <sub>12</sub> N <sub>2</sub> NiO <sub>5</sub> 394.99	Yellow greenish	188	71	55.09 (54.73)	3.32 (3.06)	7.45 (7.09)	15.51 (14.86)
[CuHL(H <sub>2</sub> O)]	C <sub>18</sub> H <sub>12</sub> CuN <sub>2</sub> O <sub>5</sub> 399.84	Yellow greenish	190	82	55.00 (54.07)	3.32 (3.02)	7.53 (7.01)	14.99 (15.89)
[ZnHL(H <sub>2</sub> O)]	C <sub>18</sub> H <sub>12</sub> ZnN <sub>2</sub> O <sub>5</sub> 401.69	Light Brown	199	88	54.00 (53.82)	3.32 (3.01)	7.53 (6.97)	15.99 (16.28)
[PdHL(H <sub>2</sub> O)]	C <sub>18</sub> H <sub>12</sub> N <sub>2</sub> O <sub>5</sub> Pd 442.72	Brown	182d	67	49.04 (48.83)	2.21 (2.73)	6.62 (6.33)	25 (24.04)
[CdHL(H <sub>2</sub> O)]	C <sub>18</sub> H <sub>12</sub> CdN <sub>2</sub> O <sub>5</sub> 448.71	Light Brown	219	78	48.00 (48.18)	3.32 (2.70)	6.53 (6.24)	25.99 (25.05)
[HgHL(H <sub>2</sub> O)]	C <sub>18</sub> H <sub>12</sub> HgN <sub>2</sub> O <sub>5</sub> 536.89	Light Brown	211	74	40.00 (40.27)	3.02 (2.25)	5.53 (5.22)	36.99 (37.36)



Scheme 2. Proposed Structures of its Metal Ions Complexes

**Mass Spectra [8]:****Ligand H<sub>2</sub>L**

The electron impact spectrum of [H<sub>2</sub>L] confirms the probable formula by showing a peak at 320.08 m/z, corresponding to complex moiety [(C<sub>18</sub>H<sub>12</sub>N<sub>2</sub>O<sub>4</sub>), calculated atomic mass 320]. The series of peaks in the range of 319, 302, 288, 261, 196 and 143 m/z may be assigned to various fragments.

**Complex of ZnL**

The electron impact spectrum of [ZnL(H<sub>2</sub>O)] confirms the probable formula by showing a peak at 401.7 m/z, corresponding to complex moiety [(C<sub>18</sub>H<sub>12</sub>N<sub>2</sub>O<sub>5</sub>Zn), calculated atomic mass 401]. The series of peaks in the range of 383.7, 280, 170, 114 and 106

m/z may be assigned to various fragments.

### Complex of HgL

The electron impact spectrum of [Hg L(H<sub>2</sub>O)] confirms the probable formula by showing a peak at 536.9 m/z, corresponding to complex moiety [(C<sub>18</sub>H<sub>12</sub>N<sub>2</sub>O<sub>5</sub>Hg), calculated atomic mass 536.87]. The series of peaks in the range of 518.9, 398.8, 216.6, 184.2 and 122 m/z may be assigned to various fragments.

### Complex of MnL

The electron impact spectrum of [MnL(H<sub>2</sub>O)] confirms the probable formula by showing a peak at 391 m/z, corresponding to complex moiety [(C<sub>18</sub>H<sub>12</sub>N<sub>2</sub>O<sub>5</sub>Mn), calculated atomic mass 291.24]. The series of peaks in the range of 373.22, 218.11 and 155.11 m/z may be assigned to various fragments.

### IR spectra

The IR spectra bands of the ligand(H<sub>2</sub>L) and its complexes were characterized at 3076, 2889, 3341, 1605 and 1569 cm<sup>-1</sup>

due to the  $\nu(\text{CH})$  aromatic,  $\nu(\text{CH})$  aldehyde,  $\nu(\text{O-H})$  phenol,  $\nu(\text{COOH})$  carbonyl and  $\nu(\text{N=N})$  azo, functional groups, respectively, for the ligand [9]. The IR spectra of the complexes exhibited ligand bands with the appropriate shifts due to complex formation [10]. On complex formation the bands of  $\nu(\text{N=N})$  and  $\nu(\text{COO})$  are shifted to lower frequencies by (11 to 77) and (19 to 41) cm<sup>-1</sup> respectively; these shifts confirm the coordination of the ligand through the nitrogen of the azo group and the carboxylate ion with the metal ions. The presence of coordination water in the spectra of all complexes were suggested by the very broad absorption around (3513 to 3393) and (910 to 870) cm<sup>-1</sup>. This indicates that the ligand was coordinated with the metal ions through the (O) carbonyl group, (O) phenol group and (N) azo group atoms. At lower frequency the complexes exhibited new bands around (588-480), and (450-412) cm<sup>-1</sup> assigned to the  $\nu(\text{M-N})$  and  $\nu(\text{M-O})$ , respectively [11,12].

**Table (2): FT-IR spectral data (wave number  $\nu$ ) cm<sup>-1</sup> for the ligand and its complexes**

Compounds	C-H arom	COOH	N=N	M-N	M-O	Other bands
H <sub>2</sub> L	3076	1605	1569	-	-	$\nu(\text{O-H})$ phenol = 3341
[MnHL(H <sub>2</sub> O)]	3100	1586 1340	1492	480	430 408	$\nu(\text{H}_2\text{O})$ = 3487, 890
[CoHL(H <sub>2</sub> O)]	3029	1576 1325	1548	570 508	440 410	$\nu(\text{H}_2\text{O})$ = 3513, 900
[NiHL(H <sub>2</sub> O)]	3111 3035	1581 1332	1532	588	450 433	$\nu(\text{H}_2\text{O})$ = 3393, 910
[CuHL(H <sub>2</sub> O)]	3118 3065	1580 1330	1557	554	425 413	$\nu(\text{H}_2\text{O})$ = 3464, 896
[ZnHL(H <sub>2</sub> O)]	3101 3078	1576 1333	1497	534	442 412	$\nu(\text{H}_2\text{O})$ = 3465, 894
[PdHL(H <sub>2</sub> O)]	3103	1564 1327	1544	503	432	$\nu(\text{H}_2\text{O})$ = 3443, 888
[CdHL(H <sub>2</sub> O)]	3100 3042	1570 1322	1551	555	432 449	$\nu(\text{H}_2\text{O})$ = 3393, 893
[HgHL(H <sub>2</sub> O)]	3054	1576 1326	1558	532	440 415	$\nu(\text{H}_2\text{O})$ = 3483, 870

arom = aromatic

### Electronic spectral, magnetic moments

The electronic spectrum of the ligand exhibit intense absorption at 273, 278 and 315 nm attributed to  $\pi \rightarrow \pi^*$  and

$n \rightarrow \pi^*$  respectively. The electronic spectrum of Ni(II) complex showed three broad peaks at 488, 867 and 873 nm assigned to  ${}^3T_{1(F)} \rightarrow {}^3A_{2(F)}$ ,  ${}^3T_{1(F)} \rightarrow {}^3T_{1(P)}$  and  ${}^3T_{1(F)} \rightarrow {}^3T_{2(F)}$

respectively, suggesting an tetrahedral geometry. The electronic spectrum of Pd(II) complex showed two broad peaks at 493 and 765 nm assigned to  $^1A_{1g} \rightarrow ^1B_{1g}$  and  $^1A_{1g} \rightarrow ^1A_{2g}$ , suggesting an square planar geometry, the electronic spectrum of Mn(II) complex showed two broad peaks at 418 and 568 nm assigned to  $^6A_1 \rightarrow ^4T_2(G)$  and  $^6A_1 \rightarrow ^4T_1(G)$  respectively, suggesting an tetrahedral geometry [9, 13]. The Co(II) complex, there are three absorption bands, two in the visible region at 431 and 604 nm which are attributed to the  $^4A_{2(F)} \rightarrow ^4T_{1(F)}$  and  $^4A_{2(F)} \rightarrow ^4T_{2(F)}$  respectively, suggesting an tetrahedral geometry. The yellow greenish of the Cu(II) complex show only band in the visible region of the lower energy was

attributed to  $^2T_2(D) \rightarrow ^2E(D)$ . In fact, it is difficult to recognize between a square planer and tetrahedral Cu(II) complex because it occurs in the same region for both geometries[14-16].

In this case the magnetic moment for Cu(II) complex is 1.901 B.M which confirmed the tetrahedral geometry for the [CuHL(H<sub>2</sub>O)] complex. Finally, the absorption bands of the Zn(II), Cd(II) and Hg(II) complexes exhibited charge transfer at 335, 299 and 300 nm respectively. This is because of the electronic configuration and the diamagnetic of these complexes which confirmed the absence of any d-d electronic transition [16]. All the absorption bands were fully assigned in Table(3).

**Table (3): Electronic data magnetic moment and molar conductivity for the metal complexes**

Complexes	Molar Conductive S. cm <sup>2</sup> mole <sup>-1</sup> in DMSO	$\mu_{\text{eff}}$ M. B.	$\lambda_{\text{max}}$ nm	$\nu$ cm <sup>-1</sup>	$\epsilon_{\text{max}}$ Lmol <sup>-1</sup> cm <sup>-1</sup>	Assignment
[MnHL(H <sub>2</sub> O)]	12	5.11	270 418 568	37037 23923 17605	419 581 202	Charge transfer $^6A_1 \rightarrow ^4T_{2(G)}$ $^6A_1 \rightarrow ^4T_{1(G)}$
[CoHL(H <sub>2</sub> O)]	14	3.88	310 431 604	32258 23201 16556	218 58 93	$^4A_{2(F)} \rightarrow ^4T_{1(F)}$ $^4A_{2(F)} \rightarrow ^4T_{1(F)}$ $^4A_{2(F)} \rightarrow ^4T_{2(F)}$
[NiHL(H <sub>2</sub> O)]	6	2.87	300 488 867 873	33333 20491 11534 11454	207 67 39 41	Charge transfer $^3T_{1(F)} \rightarrow ^3A_{2(F)}$ $^3T_{1(F)} \rightarrow ^3T_{1(F)}$ $^3T_{1(F)} \rightarrow ^3T_{2(F)}$
[CuHL(H <sub>2</sub> O)]	14	1.901	295 464	33898 21551	282 47	Charge transfer $^2T_2(D) \rightarrow ^2E(D)$
[ZnHL(H <sub>2</sub> O)]	19	dia	335	29850	337	Charge transfer
[PdHL(H <sub>2</sub> O)]	9	dia	270 493 765	37037 20284 13072	114 60 10	Charge transfer $^1A_{1g} \rightarrow ^1B_{1g}$ $^1A_{1g} \rightarrow ^1A_{2g}$
[CdHL(H <sub>2</sub> O)]	10	dia	299	33444	160	Charge transfer
[HgHL(H <sub>2</sub> O)]	11	dia	300	33333	207	Charge transfer

dia= diamagnetic

### Theoretical study

The vibration spectra of the ligand and complexes were calculated by using a semi-empirical (PM3) method. The results obtained for wave numbers are presented in, Figure (1) and the comparison with the experimental values indicates some deviations. These deviations may be due to the harmonic oscillator approximation and lack of electron correlation. It was reported [16]

that frequencies coupled with Hartree-Fock Theory (HFT) approximation and a quantum harmonic oscillator approximation tends to be 10% too high.

### Electrostatic Potential (E.P)

Electron distribution governs the electrostatic potential of molecules [17] and describes the interaction of energy of the molecular system with a positive point charge, so it is useful for finding sites of reaction in a molecular positive

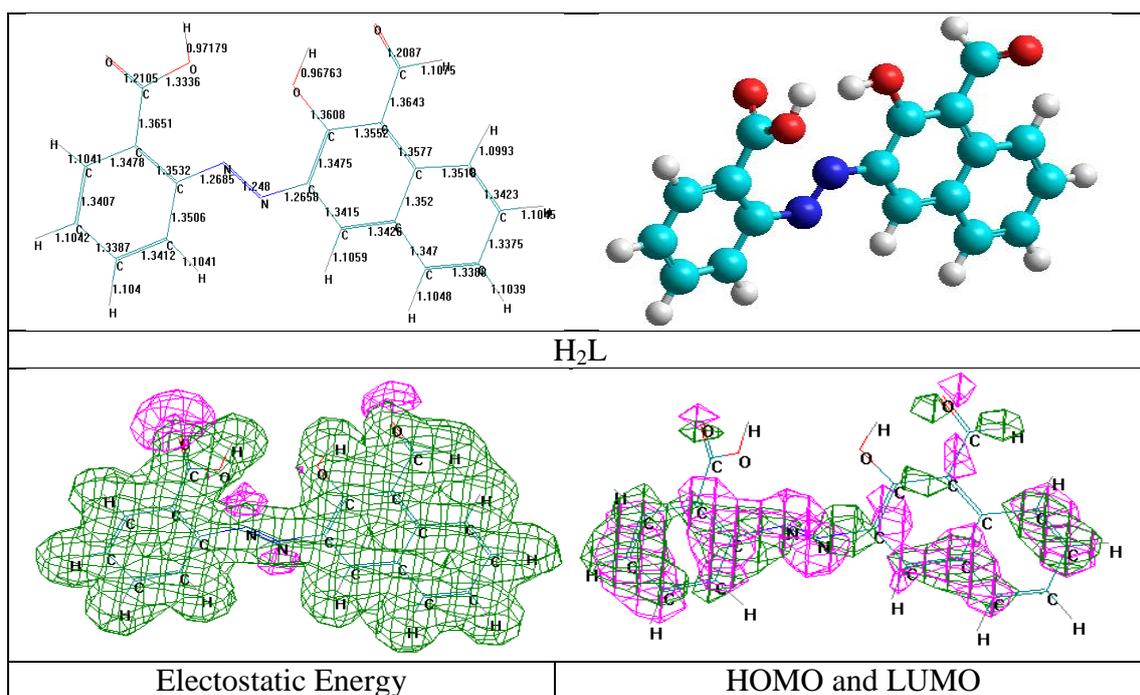
charged species tend to attack a molecule where the E.P. is strongly negative electrophilic attach. The E.P of free ligand were calculated and plotted as D contour to investigate the reactive sites of the molecules, and one can interpret the stereochemistry and rates of many reactive in involving soft electrophiles and nucleophiles in terms of the properties of frontier orbitals (HOMO and LUMO). Overlap between the HOMO and LUMO values were plotted as D contour to get more information about these molecules Figure (1). The results of calculation showed that the LUMO of metal ions prefers to react with the HOMO of nitrogen atoms of ligand.

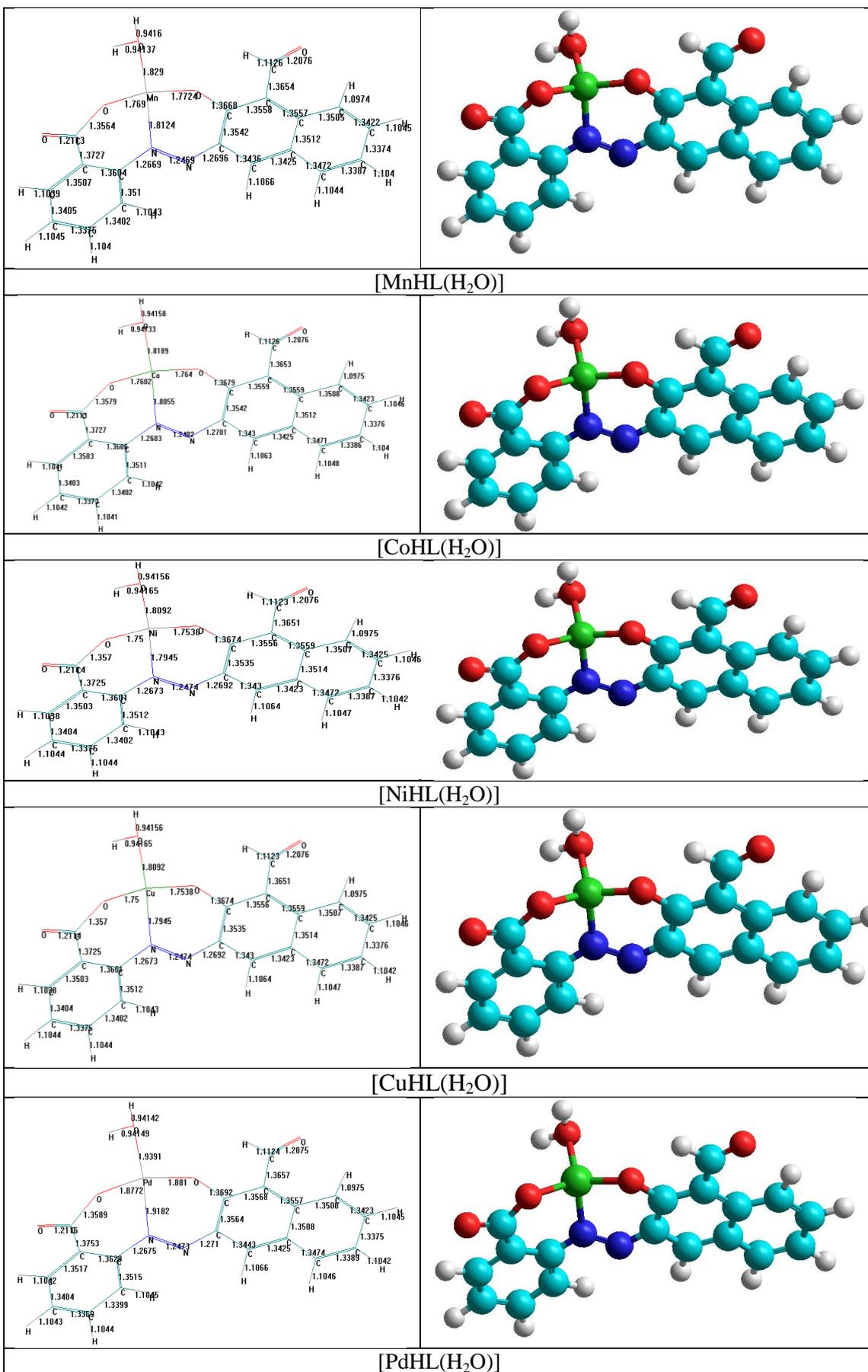
### Optimized Geometries Energy of Compounds

A theoretically probable structure of a metal complexes with ligand were calculated to search for the most probable model building stable structure, these shapes, show the calculated optima geometries for ligand. The results of PM3 and ZINDO method of calculation in gas phase for the binding energies and heat of formation of complexes are described in Table 4. The conformation structures and bond lengths of the ligand and its complexes showed in Figure (1).

**Table (4): Conformation energetic in (K J.Mol<sup>-1</sup>) and dipole moment for ligand and their metal complexes**

Compounds	Binding Energy	Heat of Formation	Electronic Energy	Dipole moment (debye)	Total Energy
L	-4219.1508260	-53.6708260	-620953.6099810	4.955	-87967.7551580
MnL	-4463.7919291	-171.0529291	-757596.1913254	6.532	-103773.8206951
CoL	-4638.8927885	-311.4537885	-812140.7141315	3.778	-112992.1543385
NiL	-4614.1299383	-286.2909383	-820670.5855286	4.478	-118832.8415333
CuL	-4458.2862378	-152.5472378	-817709.1677352	4.174	-122087.3738178
ZnL	-13804.1677715	-9203.6947715	-1221553.4295036	6.309	-197904.2944350
PdL	-4445.3205293	-130.2815293	-796851.2520453	5.04	-118768.8674303
CdL	-4184.1247524	67.6342476	-669862.1343952	5.745	-95122.9643964
HgL	-91087.5830940	-1.9893096	-686895.9947591	7.003	-95329.3014036







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Spectrochimica Acta Part A: Mole.and Biomol., 137: 75-89.

## تحضير ودراسة طيفية لبعض معقدات ايونات المعادن للـ 2-((4-فورمايل-3-هيدروكسي نفتالين-2-ايل) ثنائي زنييل) بنزويك اسد

ناصر شعلان  
زينب عبد الهادي حسن

عباس علي صالح الحمداني  
سحر صبيح حسن

قسم الكيمياء، كلية العلوم للبنات، جامعة بغداد. العراق

### الخلاصة

حضر ليكاند أزو جديد  $2-((4\text{-formyl-3-hydroxynaphthalen-2-yl)diazenyl)benzoic acid}$  من تفاعل 2- امينو حامض البنزويك مع 2- هيدروكسي-1-نفتالديهايد. حضرت معقدات الليكاند بالصيغة العامة  $[M^{II}(L)(H_2O)]$  حيث  $M =$  (منغنيز، كوبلت، نيكل، نحاس، خارصين، بلاديوم، كاديوم والزنك) بتكافؤهم الثنائي. عزلت وشخصت المركبات المحضرة بواسطة تقنيات الأشعة تحت الحمراء، وفوق البنفسجية – المرئية وطيف الرنين النووي المغناطيسي للبروتون ودرست اطياف الكتلة والحساسية المغناطيسية والتحليل الدقيق للعناصر محتوي الكلور ونسبة المعادن والتوصيلية المولارية. أظهرت الدراسة ان جميع المعقدات ذات شكل رباعي السطوح فقط معقد البلاديوم ذو شكل مربع مستوي درست النسبة المولية للمعدن التي اعطت نسبة (1:1) (فلز: ليكاند). أجريت المعالجة النظرية للمركبات المحضرة في الطور الغازي باستخدام برنامج Hyper Chem-8

الكلمات المفتاحية: أصباغ الأزو، 2-هيدروكسي-1-نفتالديهايد و معقدات الأزو