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Chelation Behaviour of New Tetradentate Schiff Base Type N₂O₂ Derived from Benzil and O-aminophenol Towards VO(II), Mn(II), Co(II), Ni(II), Cu(II), Zn(II), Pd(II) and Cd(II) Ions.

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Abstract

Benzil was reacted with 2-amino phenol in methanol as a solvent in presence of drops of glacial acetic acid as a catalyst, to give a new Schiff base as tetradentate ligand [H₂L], (C₂₆H₂₀N₂O₂) containing N and O donor atoms type (N₂O₂) : 2-((1E)-2-[2-hydroxyphenyl]imino)-1,2-diphenylethylidene}amino)phenol. This ligand [H₂L] was reacted with some metal ions in methanol to give a series of new metal complexes of two general formula : [ML] and [M'L].H₂O. (where M= VO(II), Co(II), Ni(II), Cu(II), Pd(II) and Cd(II); M' = Mn(II) and Zn(II), L= C₂₆H₁₈N₂O₂). All compounds were characterized by spectroscopic methods (IR, U.V-Vis, atomic absorption), microanalysis of the elements C.H.N, magnetic susceptibility along with conductivity measurements . From the above data the suggested geometrical structure from Mn(II), Co(II), Ni(II), Cu(II), Zn(II) and Cd(II) complexes is tetrahedral geometry, while Pd(II) and VO(II) form square planer and square pyramidal geometries respectively.

Keyword :- Benzil , 2-amino phenol , diphenylethylidene , Schiff base.

Introduction

Generally, the chelating ligands are poly functional molecules which can encage heavy metals in an organic sphere. Many types of Schiff base ligands are known and the properties of their metal complexes have been investigated⁽¹⁻³⁾. A cyclic ligands with a variety donor sets such as (N,N), (N,S), and (N,O) in their structure can act as effective chelating agents for transition and non transition metal ions⁽⁴⁾. The increasing proportion of the application and utilization of these compounds as agents has given rise to an intensification in to the investigation of the complexes, especially vic-dioxime compounds, in technique, owing to resemble of the vic-dioxime to vitamin B₁₂ and chlorophylls as a colour material of the plants, the importance of the illuminate of the biological structure have increased^(5,6). Many copper complexes are used as anti- inflammatory, anti- arthritic, anti-ulcer, anti -convulsant and anti- tumour agents^(7,8). Finally, many Co(II), Ni(II), Cu(II) and Zn(II) Schiff base complexes have been reported, the geometry of these complexes depend on the ligand donor set^(9,10). The present paper reports synthesis and characterization of new Schiff base ligand 2-((1E)-2-[2-hydroxyphenyl]imino)-1,2-diphenylethylidene}amino)phenol, from reaction

benzil with 2-aminophenol and its complexes with VO(II), Co(II), Ni(II), Cu(II), Zn(II), Pd(II) and Cd(II) metal ions.

Experimental

Materials and instrumentation

All chemicals are from fluka and reagent- dehydrate companies . Elemental microanalysis were carried out by C.H.N analyzer, model 1106 (Calo-Ebra). Metal contents of the complexes were determined by shimadzu -A.A- 680G atomic absorption spectrophotometer. IR spectra were recorded as KBr discs by using shimadzu 8300 FT-IR spectrophotometer in the range (4000-400) cm⁻¹. Electronic spectra of the prepared compounds were measured in the range (200-900) nm for 10⁻³ M solution in DMSO at 25°C by using shimadzu 160 spectrophotometer with 1.000 ± 0.001 cm matched quartz cell. Electrical molar conductivity measurements of the complexes were recorded at 25°C for 10⁻³ M solution of samples in DMSO by using PW 526 digital conductivity meter. Melting points were recorded by using sturatt melting point apparatus.¹H NMR spectrum of ligand was recorded on Bruker AMX 400 MHz spectrometer using DMSO – d₆ as a

solvent and TMS as reference. The magnetic moments were measured with a magnetic susceptibility balance (Jonson Matty catalytic system division).

Synthesis of ligand [H₂L]

Benzil (0.21gm, 1mmole) was dissolved in methanol (30ml) in presence of a lot of drops of glacial acetic acid in (100ml) round bottom flask, a pale yellow solution formed, then 2-aminophenol (0.218gm, 2mmole) was dissolved in (25ml) of methanol, was added to the above solution with stirring. The mixture was allowed to reflux at 55°C for (8hours), then cooled at room temperature and filtered let the no reacted starting materials and methanol to be removed under vacuum to give a dark brown precipitate then recrystallized by ethanol to give dark brown crystals, yield (0.36gm), (92%) m.p. (138°-140°) C (table-1).

Synthesis of VO(II) Complex (C₁).

A mixture of the ligand [H₂L] (0.392gm, 1mmole) in methanol (25ml) was added dropwise with stirring to (100ml) round bottom flask contains a solution of VOSO₄.H₂O (0.175gm, 1mmole) in (15ml) methanol. The reaction mixture was allowed to reflux for (4hours) during which time the solution colour became olive, the solvent was evaporated under reduced pressure then olive solid was obtained, washed with dry diethylether (5ml) and dried under vacuum to give yield 84% of the title complex.

Synthesis other complexes (C₂-C₈).

A Similar method to that mentioned in preparation of VO(II) complex (C₁) was used to prepare complexes [H₂L] with Mn(II) Co(II), Ni(II), Cu(II), Zn(II), Pd(II) and Cd(II) by using metal chloride salt. (table-1) stated the quantities, reaction condition and colour of all prepared complexes (C₁-C₈).

Results and discussion

Ligand [H₂L]

The pro-ligand [H₂L] was prepared by reaction of benzil with 2-aminophenol in methanol solvent in presence of glacial acetic acid according to the general method shown in (scheme-1). The ligand dissolve in methanol, ethanol, and DMSO, some physical properties and microanalysis C.H.N of the ligand were listed in (table-2).

The IR spectrum of the ligand (fig.-1) displayed a strong band at (1041) cm⁻¹ due to νC-N, the two bands at (1234)cm⁻¹ and (765)cm⁻¹ are referred to phenolic νC-O and δC-O respectively. The broad band at (3334)cm⁻¹ is refer to phenolic νO-H⁽¹¹⁾. In addition of

these bands, IR spectrum of the ligand displayed a new band at (1612)cm⁻¹ due to iminic νC=N, this is a good evidence to formation of Schiff base compound^(10,12). Data of IR spectrum of ligand were listed in (table-3). The UV-Vis spectrum for ligand (fig.-2) exhibits a high intense absorption peak at (300 nm, 33333 cm⁻¹, ε_{max}= 2022 L.mol⁻¹.cm⁻¹) due to (π→π*) electronic transition while the shoulder at (348, 28735 cm⁻¹, ε_{max} = 1813 L.mol⁻¹.cm⁻¹) is attributed to n→π* electronic transition⁽¹³⁾. The data of electronic spectrum for ligand were listed in (table -4). The ¹H NMR spectrum of ligand (Fig.3) which was recorded in DMSO -d₆ solvent showed the following signals :the signal at δ 12.98 ppm (2H) was attributed to the two phenolic (OH)Groups. The multiplet signals obtained in the range δ (6.88-7.52) ppm were due to the aromatic protons, while the doublet signal at δ (2.38,2.50) ppm referred to DMSO and the signal at δ 2.43 ppm referred to water molecules.

Complexes (C₁-C₈)

The reaction of ligand [H₂L] with VOSO₄.H₂O or with metal chloride salt of Mn(II) Co(II), Ni(II), Cu(II), Zn(II), Pd(II) and Cd(II) were carried out in methanol solvent under reflux (scheme-2). All complexes (C₁-C₈) are stable in solution (table-4) and they dissolve in ethanol and DMSO solvents.

On the basis of elemental analysis data (table-2) the molecular formula for prepared complexes was assigned to be [VOL], [MnL].H₂O, [CoL], [NiL], [CuL], [ZnL].H₂O, [PdL] and [CdL]. (where L= C₂₆H₁₈N₂O₂). The suggested molecular formula was also supported by spectral measurement as well as molar conductivity and magnetic moment.

Molar conductivity

The molar conductivity of the prepared complexes (C₁-C₈) in DMSO solvent in 10⁻³ M at 298° K (table-2) indicated non electrolytic nature⁽¹⁴⁾.

Magnetic moment

The values of measured magnetic susceptibility and effective magnetic moment (μ_{eff}) for the prepared complexes (C₁-C₈) are shown on (table-5), VO(II), Mn(II) Co(II), Ni(II) and Cu(II) complexes exhibit μ_{eff}. (1.92, 5.46, 4.68, 3.85 and 1.96) BM respectively which can be a normal values for high spin complexes compared with what have been found in the literature⁽¹⁵⁾. While μ_{eff} for Zn(II), Pd(II) and Cd(II) complexes showed that the complexes to be diamagnetic⁽¹⁶⁾.

Atomic absorption

The atomic absorption measurements (table-1) for all complexes gave approximated values when its comparison with theoretical values.

IR spectra

The IR spectra for all complexes (C₁-C₈) gave a different spectra in comparison with that of free ligand [H₂L]. The IR spectra of complexes are listed in (table-3). The IR spectra of complexes C₁ (fig.-4), C₂, C₃, C₄ and C₇ exhibit a shift in iminic ν C=N to higher frequency and appear at range (1628-1670) cm⁻¹ when it comparison with that of free ligand⁽¹⁷⁾. This shift to higher frequency in the position of iminic ν C=N suggests a weak coordination between N atom of iminic group (C=N) and metal ions, while the I.R. spectra of complexes (C₅, C₆) (fig.5) and (C₈) exhibit a shift in iminic ν C=N to lower frequency and appeared at range (1600-1605)cm⁻¹ when it comparison with that in free ligand, this shift to lower frequency in the position of iminic ν C=N can be related to the delocalization of metal ion electronic density in π – orbital of the ligand and formation of π – back bonding ($d\pi - p\pi$) which indicates the coordination between N atoms of iminic C=N groups of ligand and metal ion⁽¹⁸⁾. Also the spectra exhibit a shift in phenolic ν C-O to a lower frequency and appeared at range (1172-1228)cm⁻¹ in IR spectra for all complexes when it comparison with that of free ligand, this shift in position of phenolic ν C-O suggest the coordination of ligand through O atom of phenolic group with metal ions⁽¹⁹⁾. A broad band was observed at (3383) cm⁻¹ and (3387) cm⁻¹ in spectrum of Mn(II) and Zn(II) complexes assigned to ν O-H and suggested the presence of H₂O hydrated in crystal lattice of these complexes⁽²⁰⁾. While IR spectrum of VO(II) complex (C₁) appeared a band at (986)cm⁻¹ refer to ν V=O⁽²¹⁾. The above observation were further indicate by the appearance of new bands in IR spectra of all complexes at (563-586)cm⁻¹ and at (456-530)cm⁻¹ due to ν M-N and ν M-O respectively^(22,23).

Electronic spectra

The electronic spectral data for all complexes (C₁-C₈) are summarized in (table-4). The peak at (300)nm in the electronic spectrum of the ligand was shifted to higher frequency in the electronic spectra of all complexes and appeared at range (304-320)nm, these shifting in ligand field peak indicate coordination between ligand with metal ion. New absorption peak appeared at rang (395-445)nm in the electronic spectra of all complexes attributed to charge transfer electronic transition M→L⁽¹³⁾. The electronic spectrum of VO(II) complex (C₁) showed a new two

peaks (Fig.6), the first with high intensity is overlap with charge transfer peak appeared at (418)nm which was referred to (d-d) electronic transition type ${}^2B_2 \rightarrow {}^2A_1$, while the second peak at (630)nm was assigned to (d-d) electronic transition type ${}^2B_2 \rightarrow {}^2B_1$, these two absorption peaks related to square pyramidal VO(II) complexes⁽²⁴⁾. The electronic spectra of Mn(II) (C₂), Co(II) (C₃) (Fig.7), Ni(II) (C₄) and Cu(II) (C₅) complexes displayed new absorption peaks assigned to (d-d) electronic transition as follows : Mn(II) (C₂) at (422) nm (${}^6A_1 \rightarrow {}^4E, {}^4A_{1(g)}$) overlap with charge transfer peak ; Co(II) (C₃) at (652) nm (${}^4A_2 \rightarrow {}^4T_{1(p)}$); Ni(II) (C₄) at (438) nm (${}^3T_1 \rightarrow {}^1T_2$) and at (510) nm (${}^3T_1 \rightarrow {}^3T_{1(p)}$); Cu(II) (C₅) at (370) nm (${}^2B_2 \rightarrow {}^2E$), in fact these results are in good agreement with previous works of Mn(II), Co(II), Ni(II) and Cu(II) complexes of tetrahedral geometry^(24,25). While the UV-Vis spectrum of Pd(II) complex (C₇) shows a new absorption peak at (395) nm with high intensity which overlap with charge transfer peak, refer to (d-d) electronic transition type (${}^1A_{1g} \rightarrow {}^1B_{1g}$) this is a good evidence for square planer geometry about Pd(II) ion⁽²⁶⁾. Finally, the UV-Vis spectra of Zn(II) complex (C₆) and Cd(II) complex (C₈) showed no absorption peak at range (438-900) nm, that is indicate no(d-d) electronic transition happened (d¹⁰-system) in visible region and that is a good result for Zn(II) and Cd(II) tetrahedral complexes⁽²⁷⁾.

Suggest structures

On the basis of elemental analysis, molar conductivity, magnetic moment and spectroscopic studies (IR, U.V-Vis, and atomic absorption) for the ligand [H₂L] and all prepared complexes (C₁-C₈), we suggest that the ligand behaves as tetradentate on complexation with metal ions via iminic N atoms and phenolic O atoms. The following structural formula for the prepared complexes can be suggested:

1. Square pyramidal geometry for [VOL] and by depended on bonds length and angle in a similar systems (table-6) that the structure formula was drawn (Fig.-8).
2. Tetrahedral geometry for [MnL].H₂O and [CoL], [NiL], [CuL], [ZnL].H₂O and [CdL], by depended on the bonds length and angle in a similar systems (table-7) that the structure formula for [ZnL].H₂O was drawn (Fig.-9).
3. Square planer geometry for [PdL] and by depended on bonds length and angle in a similar systems (table-8) that the structures formula for this complex was drawn (Fig.-10).

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Table-1: Some physical properties of prepared compounds and weight of metal salt.

Compounds*	Colour	Yield%	Metal salt	Weight (g) = 1 mmole
H ₂ L	Brown	92	-----	-----
[VOL]	Olive	84	VOSO ₄ .H ₂ O	0.175
[MnL].H ₂ O	Pale Brown	73	MnCl ₂ .4H ₂ O	0.198
[CoL]	Reddish Brown	74	CoCl ₂ .6H ₂ O	0.236
[NiL]	Greenish Brown	71	NiCl ₂ .6H ₂ O	0.238
[CuL]	Reddish Brown	90	CuCl ₂ .2H ₂ O	0.171
[ZnL].H ₂ O	Pale Brown	76	ZnCl ₂	0.137
[PdL]	Pale Brown	64	PdCl ₂	0.178
[CdL]	Yellowish Brown	92	CdCl ₂ .H ₂ O	0.202

*L= C₂₆H₁₈N₂O₂

Table-2: Results of elemental analysis and molar conductance for prepared compounds.

Compound	Molecular Formula	M.wt	Molar conductance $\text{Ohm}^{-1} \cdot \text{cm}^2 \cdot \text{mol}^{-1}$	m.p $^{\circ}\text{C}$	Found.%,(Calc.)%			
					C	H	N	Metal
H ₂ L	C ₂₆ H ₂₀ N ₂ O ₂	392	-----	138-140	79.12 (79.59)	4.86 (5.10)	6.78 (7.14)	
C ₁	[VO(L)]	451	9.19	146-148	68.28 (69.17)	3.02 (3.99)	5.34 (6.20)	9.24 (9.97)
C ₂	[Mn(L)].H ₂ O	463	10.04	150-151	65.98 (67.38)	3.62 (4.31)	5.16 (6.04)	11.02 (11.87)
C ₃	[Co(L)]	447	9.07	160-162	68.91 (69.79)	3.28 (4.02)	5.42 (6.26)	11.94 (12.75)
C ₄	[Ni(L)]	448.7	1.43	150-152	68.91 (69.53)	3.42 (4.01)	5.19 (6.24)	12.74 (13.08)
C ₅	[Cu(L)]	453.5	9.29	164-166	68.08 (68.79)	3.12 (3.96)	5.48 (6.17)	13.48 (14.00)
C ₆	[Zn(L)].H ₂ O	473.4	5.05	150-152	65.41 (65.90)	3.76 (4.22)	5.18 (5.91)	13.18 (13.81)
C ₇	[Pd(L)]	496.4	6.39	148-150	61.98 (62.85)	2.98 (3.62)	4.96 (5.64)	20.86 (21.43)
C ₈	[Cd(L)]	502.4	5.42	160-162	61.34 (62.10)	2.86 (3.58)	4.76 (5.57)	21.72 (22.29)

Table -3 : I.R Spectral data for free ligand and prepared compounds (cm^{-1}).

Symbol of Compound	ν C=N	ν O-H	ν C-N	ν C-O	δ C-O	ν M-N	ν M-O
[H ₂ L]	1612	3334	1041	1234	756	-	-
C ₁	1659	-	1037	1211	760	582	470
C ₂	1628	3383	1035	1215	750	572	465
C ₃	1662	-	1028	1212	760	583	470
C ₄	1628	-	1037	1175	756	563	458
C ₅	1600	-	1033	1172	748	586	472
C ₆	1600	3387	1035	1215	756	565	530
C ₇	1670	-	1033	1228	756	570	458
C ₈	1605	-	1033	1172	756	572	456

Table- 4 : electronic spectral data of ligand and its complexes.

Complexes	Metal Ions	Unpaired electron	$(X_g) \cdot 10^{-6}$	$(X_M) \cdot 10^{-6}$	$(D) \cdot 10^{-6}$	$(X_A) \cdot 10^{-6}$	$\mu_{\text{eff. BM}}$
C ₁	VO ⁺²	1	2.94	1325.76	225.84	1551.6	1.92
C ₂	Mn ⁺²	5	26.27	12163.86	225.84	12389.7	5.46
C ₃	Co ⁺²	3	20.17	9018.76	225.84	9244.6	4.68
C ₄	Ni ⁺²	2	13.43	6027.66	225.84	6253.5	3.85
C ₅	Cu ⁺²	1	3.08	1395.16	225.84	1621.0	1.96
C ₆	Zn ⁺²	0	0.0	-	-	-	0.0
C ₇	Pd ⁺²	0	0.0	-	-	-	0.0
C ₈	Cd ⁺²	0	0.0	-	-	-	0.0

Table-5: values of magnetic susceptibility and effective magnetic moments ($\mu_{\text{eff.}}$) of prepared complexes.

Symbol of Compound	λ (nm)	ν (cm ⁻¹)	ϵ_{max} L.M ⁻¹ . cm ⁻¹	assignment	formula
[H ₂ L]	300	33333	2022	$\pi \rightarrow \pi^*$	
	348	28735	1863	$n \rightarrow \pi^*$	
C ₁	304	32894	2324	L.F	Square Pyramide
	418	23923	2112	C.T, ${}^2B_2 \rightarrow {}^2A_1$	
	630	15873	370	${}^2B_2 \rightarrow {}^2B_1$	
C ₂	307	32573	2300	L.F	Tetrahedral
	422	23696	1179	C.T, ${}^6A_1 \rightarrow {}^4E, {}^4A_{1(G)}$	
C ₃	306	32679	2180	L.F	Tetrahedral
	445	22471	2046	C.T	
	652	15337	370	${}^4A_2 \rightarrow {}^4T_{1(P)}$	
C ₄	320	31250	2212	L.F	Tetrahedral
	438	22831	717	${}^3T_1 \rightarrow {}^1T_2$	
	510	19607	300	${}^3T_1 \rightarrow {}^3T_{1(P)}$	
C ₅	305	32786	2468	L.F	Tetrahedral
	422	23696	1503	C.T	
	370	13698	260	${}^2B_2 \rightarrow {}^2E$	
C ₆	318	31446	2231	L.F	Tetrahedral
	437	22883	719	C.T	
C ₇	310	32258	2141	L.F	Square Planer
	395	25316	1540	C.T, ${}^1A_{1g} \rightarrow {}^1B_{1g}$	
C ₈	307	32573	1900	L.F	Tetrahedral
	436	22936	306	C.T	

Table-6: bonds length and angle in a similar systems of [VOL] complex.

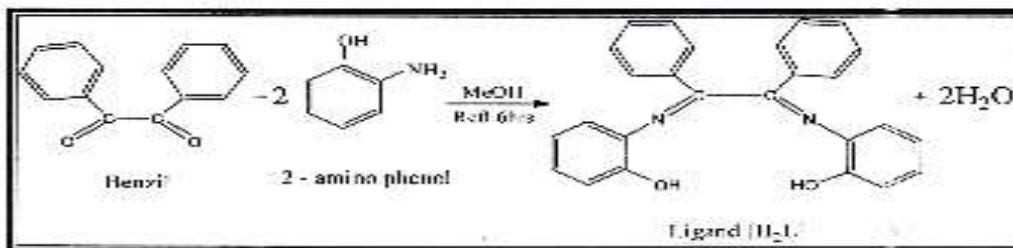
Angle ^o	Bond type	Bond length A ^o	Bond type
58.631	N ₁ -V-N ₂	1.836	V-N ₁
91.821	N ₂ -V-O ₂	1.836	V-N ₂
116.846	O ₂ -V-O ₁	1.816	V-O ₁
91.821	O ₁ -V-N ₁	1.816	V-O ₂
90	N ₁ -V=O	1.524	V=O

Table-7: bonds length and angle in a similar systems of [ZnL].H₂O complex.

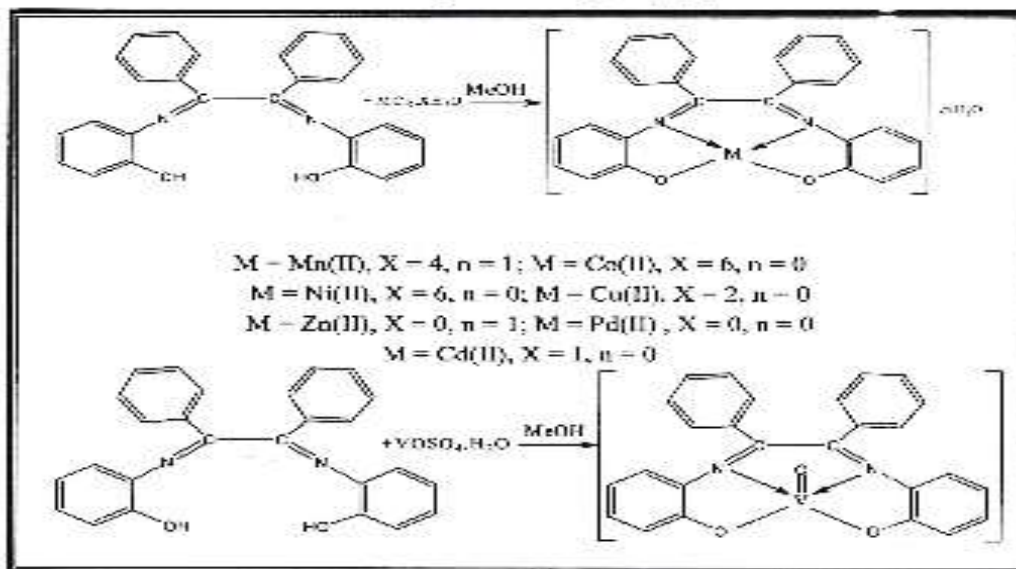
Angle ^o	Bond type	Bond length A ^o	Bond type
119.998	N ₂ -Zn-O ₁	1.926	Zn-N ₁
109.561	N ₂ -Zn-O ₂	1.926	Zn-N ₂
130.382	O ₂ -Zn-O ₁	1.890	Zn-O ₁
90	N ₁ -Zn-O ₁	1.890	Zn-O ₂
120.002	N ₁ -Zn-N ₂		
90	N ₁ -Zn-O ₂		

Table-8: bonds length and angle in similar systems of [PdL] complex.

Angle °	Bond type	Bond length Å ⁿ	Bond type
89	N ₂ -Pd-N ₃	1.882	Pd-N ₁
90	N ₂ -Pd-O ₂	1.882	Pd-N ₂
91	O ₂ -Pd-O ₁	2.0	Pd-O ₁
90	O ₂ -Pd-N ₁	2.0	Pd-O ₂



Scheme-1: synthesis of ligand [H₂L].



Scheme-2: synthesis of complexes.

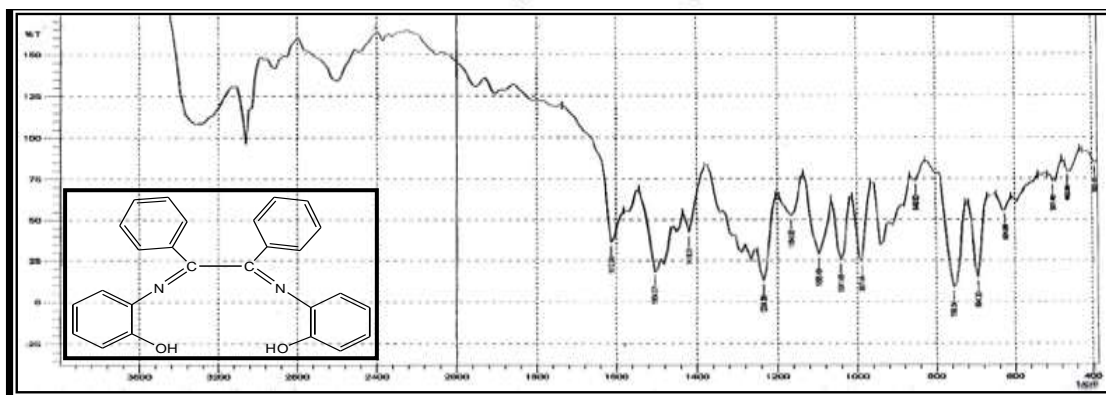


Fig.-1: The IR Spectrum of ligand [H₂L].
 2-((1E)-2-[2-hydroxyphenyl]imino)-1,2-diphenylethylidene) amino)phenol.

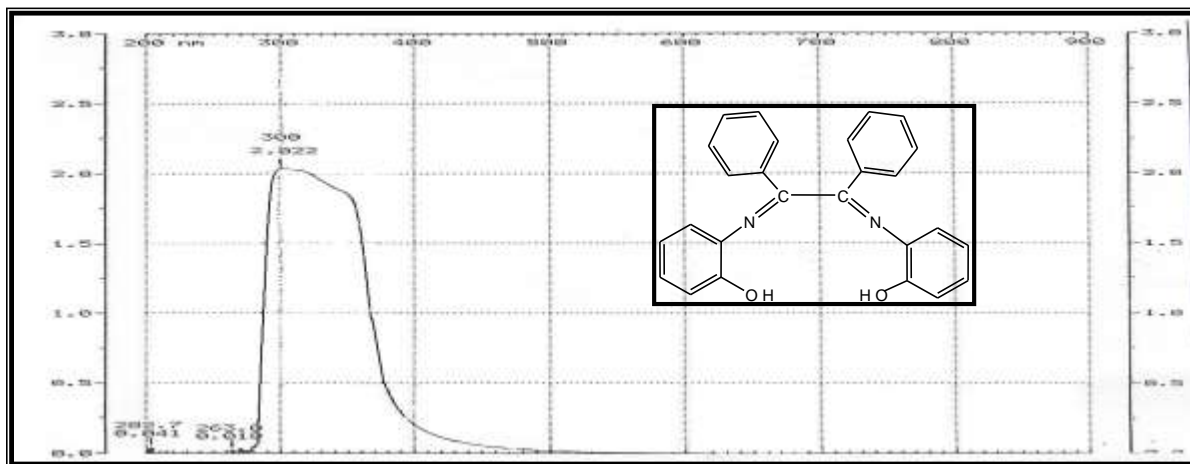


Fig.-2: The (UV-Vis) of ligand [H₂L].
2-((1E)-2-[2-hydroxyphenylimino]-1,2-diphenylethylidene) amino)phenol

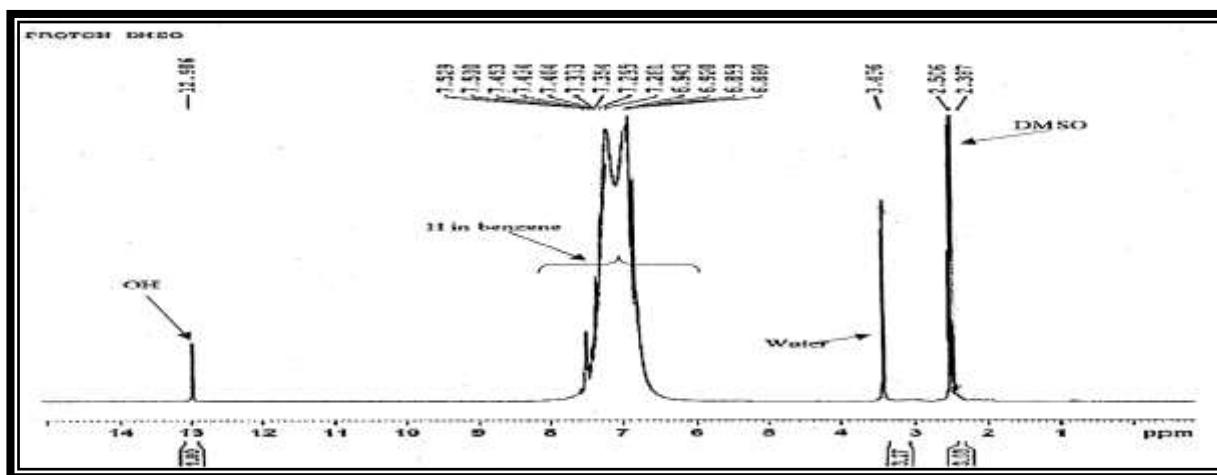


Fig.3 : ¹H NMR spectrum of ligand (H₂L)

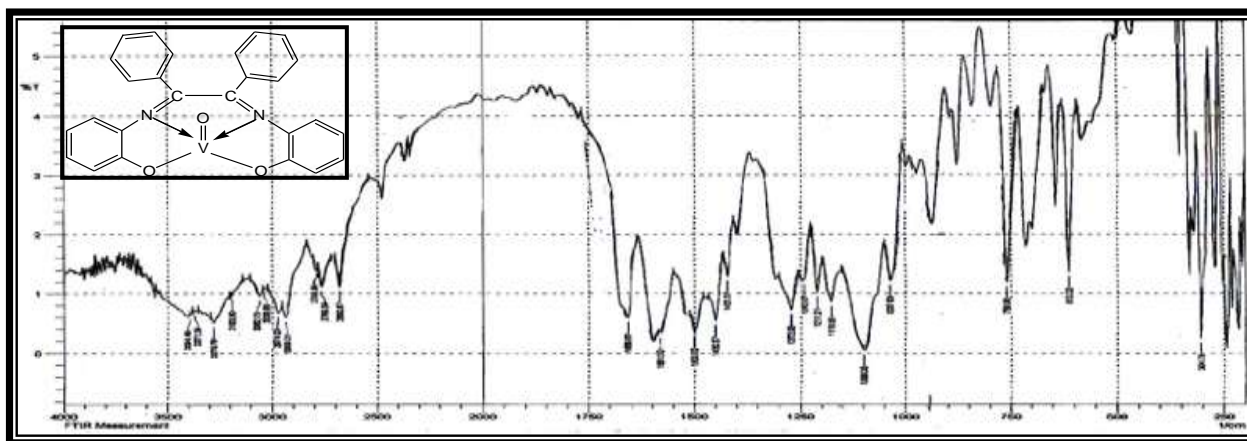


Fig.-4: I.R spectrum of [VOL] complex.

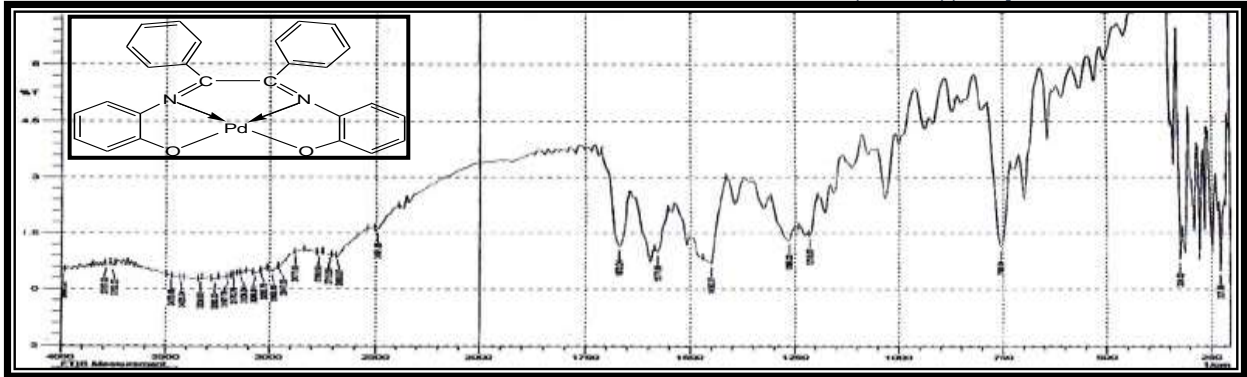


Fig.-5: I.R spectrum of [PdL] complex.

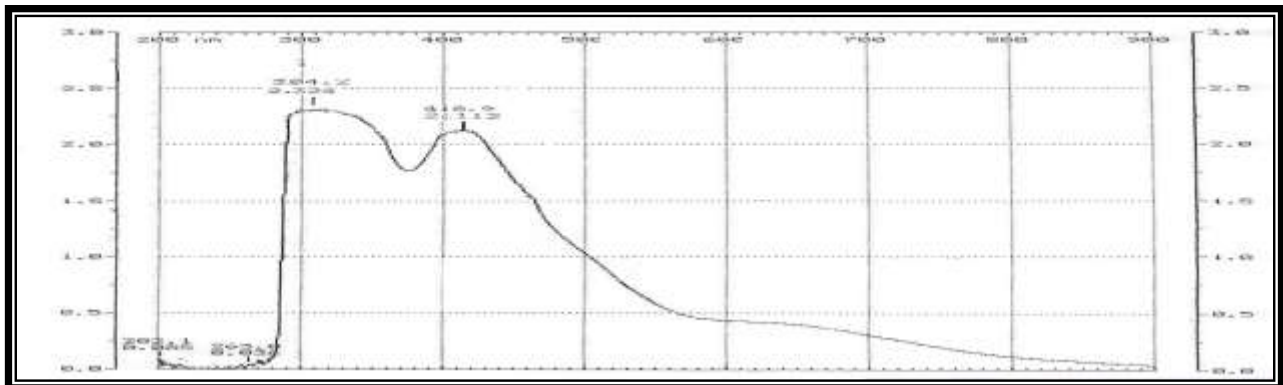


Fig.-6: (UV-Vis) spectrum of [VOL] complex.

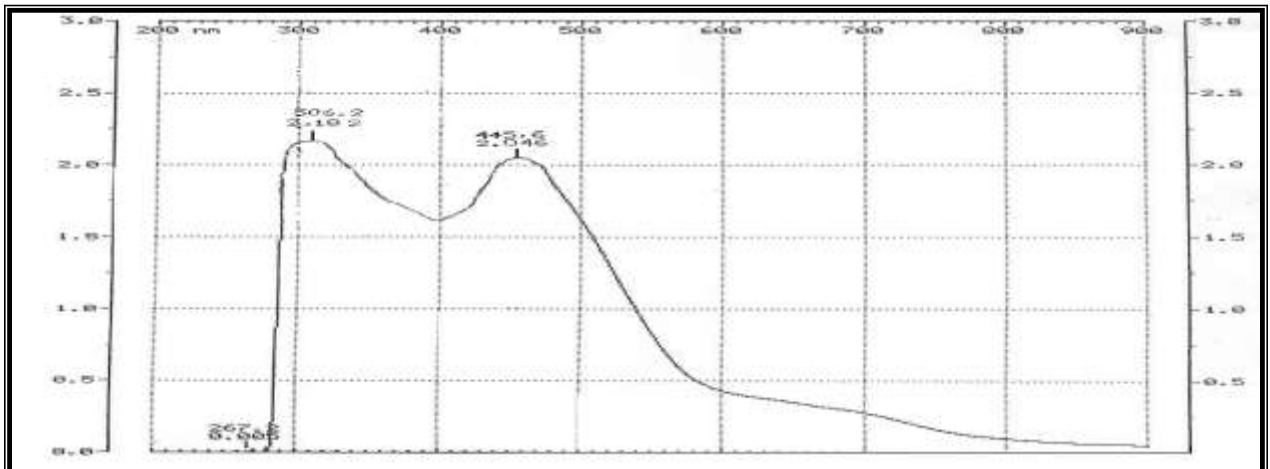


Fig.-7: (UV-Vis) spectrum of [CoL] complex.

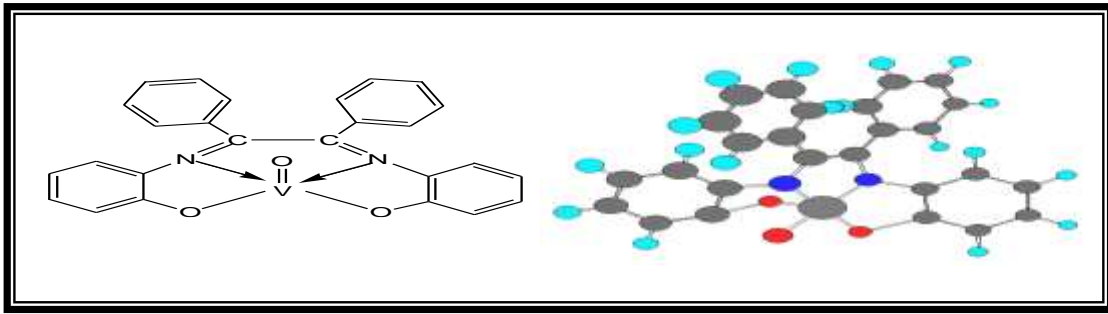


Fig.-8: proposed structure for [VOL] complex.

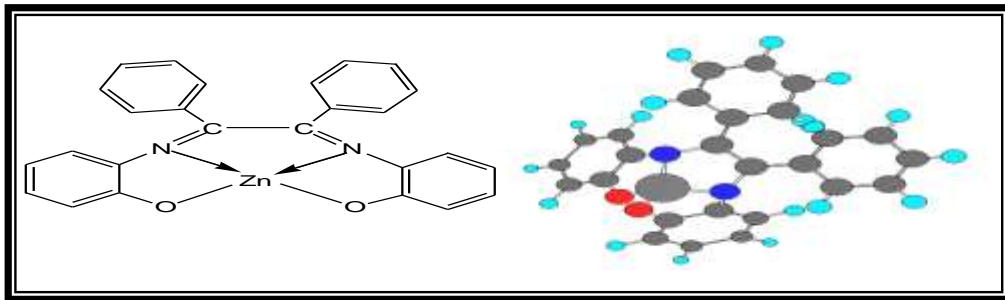


Fig.-9: proposed structure for [ZnL].H₂O complex.

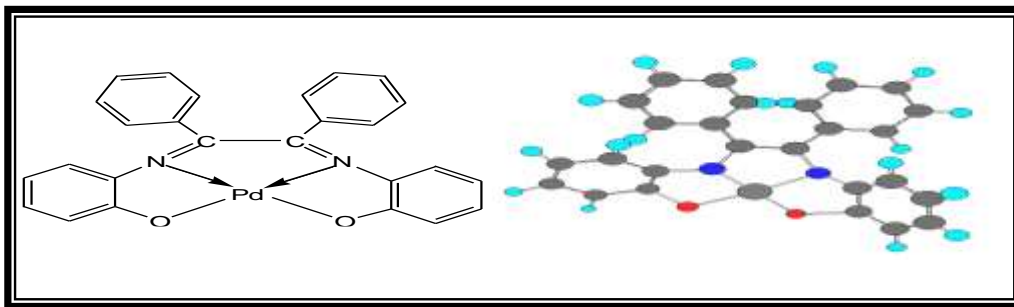


Fig.-10: proposed structure for [PdL] complex.