

Synthesis and Spectroscopic Study of New Bidentate Schiff Base type (NN) and it's Complexes with Mn^{II} , Fe^{II} , Co^{II} , Ni^{II} , Cu^{II} and Cd^{II} Ions .

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Abstract

four coordinated complexes for divalent metal ions : Mn, Fe, Co, Ni, Cu and Cd have been synthesized using bidentate Schiff base ligand type (NN) formed by the condensation of o-phenylenediamine , p-methylbenzadehyde and furfural in methanol. The ligand was reacted with divalent metal chloride forming complexes of the types $[M(L)Cl_2]$ where : $M^{II}=Mn, Fe, Ni, Cu, \text{ and } Cd$. These new compounds were characterized by elemental analysis, spectroscopic methods (FT-IR, U.V-Vis, 1H NMR (for ligand only and atomic absorption) , magnetic susceptibility, chloride content along with conductivity measurement. These studies revealed that the geometry for all complexes about central metal ion is tetrahedral.

Key words : Schiff base, o-phenylenediamine, furfural.

تحضير ودراسة طيفية لقاعدة شف جديدة ثنائية السن نوع (NN) ومعداتها مع الايونات : المنغنيز (II)، الحديد (II)، الكوبالت (II) ، النيكل (II)، النحاس (II) والكاديوم (II) .

وليد خالد مهدي ، *حيدر داود جاسم ، خولة محمد سلطان وساجد محمود لطيف

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الخلاصة

حضرت معقدات رباعية التناسق للأيونات الفلزية ثنائية التكافؤ : المنغنيز ، الحديد ، الكوبالت ، النيكل ، النحاس والكاديوم باستخدام ليكاند قاعدة شيف نوع (NN) حضرت بتكثيف اورثو-فنيولين ثنائي امين ، بارا- مثيل بنزليدهايد والفورفورال في الميثانول . ثم مفاعلة الليكاند مع ملح الفلز ثنائي التكافؤ لتكوين معقدات من نوع $[M(L)Cl_2]$ حيث يمثل $M(II)=Mn, Fe, Co, Ni, Cu \text{ and } Cd$ شخصت المركبات الجديدة بوساطة تحليل العناصر ، الطرائق الطيفية (الاشعة تحت الحمراء ، الاشعة فوق البنفسجية-المرئية ، بروتون الرنين النووي المغناطيسي(لليكاند فقط) والامتصاص الذري) ، الحساسية ، المغناطيسية ، محتوى الكلورايد فضلا عن قياسات التوصيلية . هذه الدراسات بينت ان الشكل الهندسي لجميع المعقدات حول الفلز المركزي هو رباعي السطوح .

الكلمات المفتاحية : قاعدة شيف ، اورثو-فنيولين ثنائي امين ، الفورفورال.

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Introduction

Schiff bases compounds have a great importance in coordination chemistry, due to their ability to form a range of complexes which have applications in different fields [1,2]. They are found useful in catalysis, medicine as antibiotics and anti-inflammatory agents and in the industry as anticorrosion [3-7] Schiff bases complexes containing nitrogen atoms as donor atoms play an important role in biological system and represent models for metalloproteinase and metalloenzymes [8]. Metal complexes of Schiff base have also been used in oxidation reaction [9] and for binding metal ions via the nitrogen atom lone pair, especially when used in combination with one or more donor atoms to form polydentate chelating ligands or macrocycles [10]. 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 atoms [11]. In this paper report synthesis and characterization of new schiff base ligand N-furan-2-ylmethylene-N-(4-methylbenzylidene)-benzene-1,2-diamine from reaction furfural and p-methylbenzaldehyde with 1,2-phenylenediamine and it's complexes with divalent metal ions: Mn, Fe, Co, Ni, Cu and Cd.

Experimental

Materials and physical measurement.

All chemicals are from fluka and Redial-Dehange Company. Elemental microanalysis were carried out by C.H.N analyzer, model 1106(Carlo-Erba). 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 range 1400-400 cm^{-1} . Electronic spectra of the prepared compounds were measured in range (200-900)nm for $10^{-3}M$ solution in DMSO at 25 $^{\circ}C$ by using Shimadzu 160 Spectrophotometer with 1.000 ± 0.001 cm matched quartz cell. Molar conductivity measurements of the complexes solutions were recorded at 25 $^{\circ}C$ for $10^{-3}M$ solutions of samples in DMSO by using PW 525 digital conductivity meter. Chloride was determined by using potentiometer titration method in a (686-Titro processer-665 Dosrmat-metrohm swiss). Magnetic moments were measured with a magnetic susceptibility balance (Jonson Matty Catalytic systems division). 1H NMR spectrum was acquired in DMSO- d_6 solution using a Bruker AMX400 MHz spectrometer with tetramethylsilane (TMS) as an internal standard for 1H NMR.

Preparation of ligand

The ligand N-furran-2-ylmethylene-N $^{\ominus}$ -(4-methylbenzylidene)benzene-1,2-diamine was prepared by modification of literature [12]. A mixture of 4 mmole (384mg) furfural, 4 mmole (480mg) 4-methylbenzaldehyde and 4mmole (432mg) of 1,2-phenylenediamine in 30 ml methanol was refluxed for 6hrs, the reaction mixture was cooled in ice, the pale brown product which was first formed, was filtered and washed with methanol and dried over anhydrous, calcium chloride.

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Preparation of metal complexes

All complexes were prepared by dissolving 2 mmole (456mg) of new ligand in 20 ml absolute ethanol. The solution was added gradually with stirring to 20ml solution of respective metal(II) chloride (2mmole). The mixture was stirred for 6 hrs, at 60°C. The resulting solution was evaporated under vacuum to remove. The solvent the colored solid complexes separated one in each case. The product was filtered, washed several times with ethanol and recrystallized from hot ethanol and air dried at room temperature.

Results and discussion

The pro-ligand was prepared as in scheme-1. The new ligand dissolves in DMSO and DMF, some physical properties and microanalysis C.H.N of the ligand were listed in table-1. Important infrared bands of the ligand and it's complexes together with their assignments are collected in table-2. IR spectrum of free ligand (Fig.1) displayed a band at (1111)cm⁻¹ due to ν_{C-O-C} for furan cycle[13]. The spectrum showed new bands at (1612) cm⁻¹ and (1550) cm⁻¹ assigned to $\nu_{C=N}$ (imine)[14], this is a good evidence to formation of Schiff base compound and indicating the azomethine bands are nonequivalent. In addition of these bands, the spectrum appears bands at (1010) cm⁻¹, (1267) cm⁻¹ and (1481) cm⁻¹ and (1465)cm⁻¹ due to ν_{C-N} , ν_{C-H} (aromatic), $\nu_{C=C}$ (aromatic) and ν_{CH_3} respectively [12]. The U.V-Vis spectrum of the free ligand (Fig.3) exhibits two high intense absorption peak, the first at 278 nm (35971 cm⁻¹) and the second at 299nm(33445 cm⁻¹) due to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ electronic transition respectively[14]. The data of electronic spectrum for the ligand were listed in table-3. The predicated ¹HNMR spectrum of the free ligand (Fig.5) showed peaks at δ H:8.48(1H), 8.10(1H), 7.60(4H), 7.32(4H), 6.41(2H), 5.75(1H),2.78(for DMSO-d₆ solvent) and 1.27(3H for CH_3 group)[15],these data are listed in table-4.

Complexes

The reaction of the ligand (L) with metal (II)chloride salt of Mn, Fe, Ni, Cu and Cd were carried out in solvent under reflux (scheme-1). All complexes are stable in solution (table-3) and dissolve in DMSO and DMF solvents. The molar conductivity Values for all prepared complexes in DMSO solvent in 10⁻³M at 298°K (table-1) indicated non electrolytic nature[16].On the bases of elemental analysis data (table-1), the molecular formula for prepared complexes was assigned to be [M(L)Cl₂] where: M^{II}=Mn, Fe, Ni, Cu and Cd. The suggested molecular formula was also supported by spectral measurement as well as molar conductivity and magnetic moment.

Magnetic moment

The value of effective magnetic moment μ_{eff} for he prepared complexes Mn(II),Fe(II),Ni(II), and Cu(II) are shown in (table-3), which exhibit μ_{eff} .(5.42,4.35,3.81 and 1.94) B.M respectively which can be a normal values for tetrahedral complexes compared with what have been found in the literature [17], while μ_{eff} . For Cd(II)complex showed that the complex to be diamagnetic.

Atomic absorption

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

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Chloride content

The chloride content were determined by using potentiometric titration method which showed that all prepared complexes contain chloride, this indicated the coordination between metal ions and Cl⁻ (table-1).

IR spectra

Infrared spectral data for all complexes are listed in table-2. The IR spectra for complexes gave a different spectra in comparison with that of free ligand(L). The IR spectra of all complexes (Fig.2) exhibit a shift in two bands for $\nu(\text{C}=\text{N})$ (iminic) to lower frequency and appeared as signal band (with shoulder) at range (1598-1592) cm^{-1} , when it comparison with that of free ligand, this shift can be related to the delocalization of metal ion electronic density in π -orbital of the ligand and formation π -back bonding ($d\pi$ - $p\pi$) which indicates the coordination between N atom of iminic group (C=N) and metal ion [18]. The above observation were further indicate by the appearance of new band in IR spectra of all complexes at range (594-590) cm^{-1} due to $\nu(\text{M}-\text{N})$ [12].

Electronic spectra

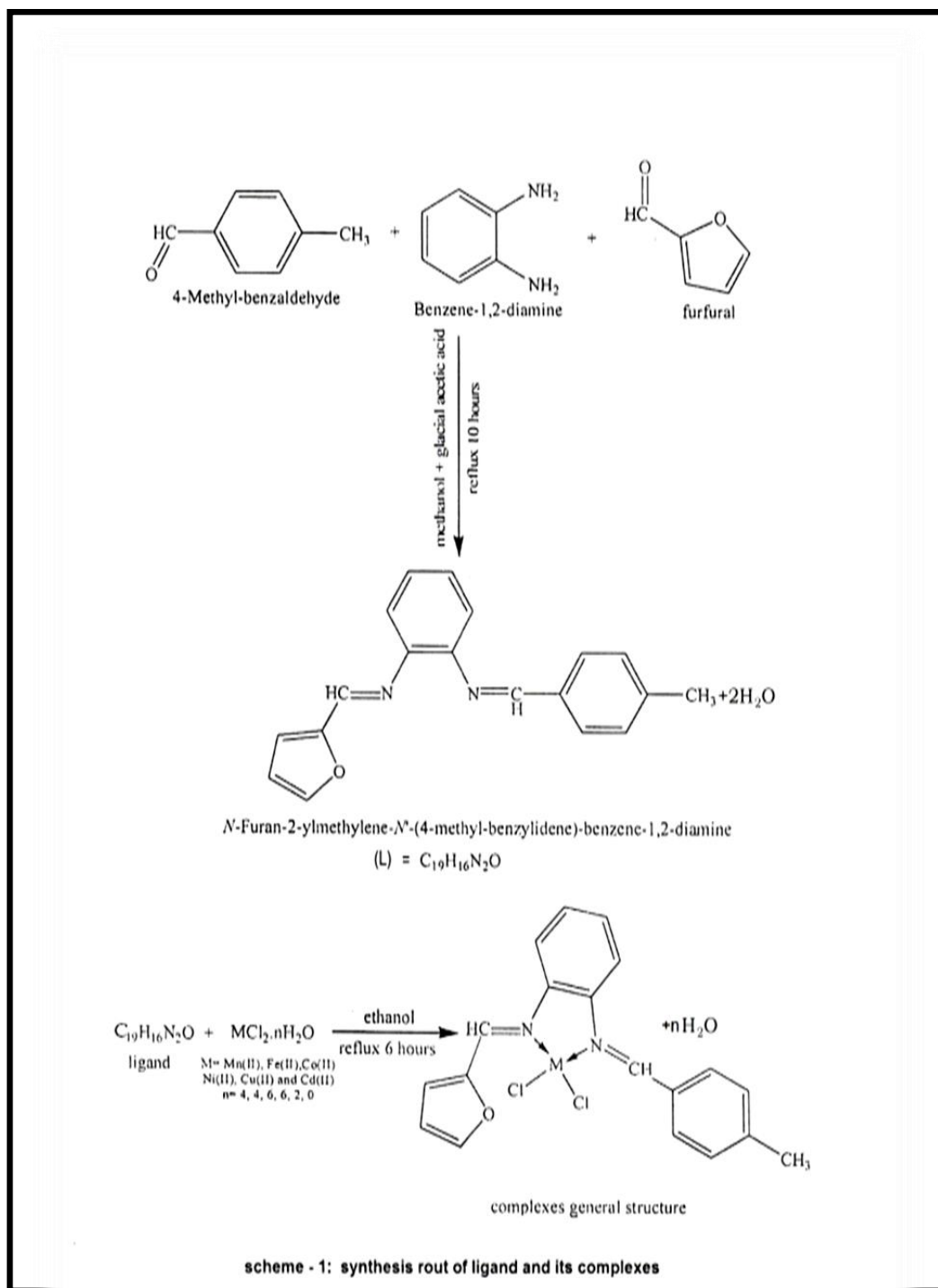
The electronic spectral data for all complexes are summarized in table-3. The electronic spectra of the prepared complexes exhibit peaks around (265-272) nm (37736-36764) cm^{-1} and at range (33445 - 33112) cm^{-1} related to $\pi \rightarrow \pi^*$ For intra ligand and new peak at range (380-396)nm (26315-25253) cm^{-1} refers to charge transfer (C.T) transition. The electronic spectra confirm the stability of the prepared complexes in DMSO solvent and the structure of complexes in solution are in accord with their structure in the solid state. The electronic spectrum of Mn(II) complexes appeared a peak at 685nm (14598) cm^{-1} refers to (d-d) electronic transition type ${}^6\text{A}_1 \rightarrow {}^4\text{T}_1$ of tetrahedral Mn(II) complex[19]. The U. V-Vis spectrum of Fe(II) complex (Fig-4) displayed new absorbance peak at 788nm (12690) cm^{-1} attributed to (d-d) electronic transition type ${}^5\text{E}_2 \rightarrow {}^5\text{T}_2$ of tetrahedral Fe(II) complex[19]. The new absorption peak in electronic spectrum of Co(II) complex at 676nm (14793) cm^{-1} can be assigned to (d-d) electronic transition type ${}^4\text{A}_2(\text{F}) \rightarrow {}^4\text{T}_2(\text{P})$ this result is a good evidence for Co(II) tetrahedral geometry [19]. The electronic spectrum of Ni(II) complex shows two new peaks with weak intensity, the first appeared at 525nm (19047) cm^{-1} and the second at 680nm (14706) cm^{-1} can be assigned to (d-d) electronic transition type ${}^3\text{T}_1(\text{F}) \rightarrow {}^3\text{T}_2(\text{F})$ and ${}^3\text{T}_1(\text{F}) \rightarrow {}^3\text{T}_1(\text{P})$ respectively, in fact this result is a good agreement with previous work of Ni(II) tetrahedral geometry[15,19]. A new absorption peak at 776nm(12886) cm^{-1} was observed in electronic spectrum of Cu(II) complex which had been attributed to (d-d) electronic transition type ${}^2\text{T}_2 \rightarrow {}^2\text{E}$, a position of this peak is a good agreement with reported for Cu(II) distortion tetrahedral geometry[20]. The U.V-Vis spectrum of Cd(II) complex exhibited a new peak at 380nm (26315) cm^{-1} attributed to charge transfer electronic transition (C.T) normally prefers tetrahedral Coordination[15].

Conclusion

On the bases of elemental analysis, molar conductivity, magnetic moment, chloride content and spectroscopic studies [FT-IR, U.V-Vis, ¹HNMR(for lignd only), Atomic Absorption] for the ligand and it's complexes, we suggest that the ligand behaves as bidentate on complexation with metal ion via two N atoms of iminic groups which formed tetrahedral geometry about divalent metal ion.

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Table-1: some physical properties and molar conductance for free ligand and it's complexes.

Compound	M.wt g.mol ⁻¹	Colour	Yield %	m.p C°	Λ _M Ohm ⁻¹ .cm ² .mol ⁻¹
Ligand(L) C ₁₉ H ₁₆ N ₂ O	288	Brown	89	198	—
[Mn(L)Cl ₂]	414	Pale Brown	78	270	9.24
[Fe(L)Cl ₂]	415	Pale Brown	75	210	6.46
[Co(L)Cl ₂]	418	Pale Red	82	274	7.68
[Ni(L)Cl ₂]	417.7	Pale Red	84	265	8.43
[Cu(L)Cl ₂]	422.5	Pale Brown	74	240	5.42
[Cd(L)Cl ₂]	471.4	Yellowish Brown	88	260	4.21

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Table-2: Elemental analysis for free ligand(L) and it's complexes.

Compound	Found % , (Calc)%				
	M	C	H	N	Cl
Ligand (L)	---	(79.16)	(5.55)	(9.72)	---
C ₁₉ H ₁₆ N ₂ O		78.64	5.08	9.24	
[Mn(L)Cl ₂]	(13.28)	(55.07)	(3.86)	(6.76)	(17.13)
	12.96	54.84	3.52	6.43	16.78
[Fe(L)Cl ₂]	(13.49)	(54.94)	(3.85)	(6.74)	(17.10)
	12.78	54.16	3.24	5.92	16.82
[Co(L)Cl ₂]	(14.11)	(54.54)	(3.82)	(6.69)	(16.98)
	13.63	54.17	3.18	5.94	16.46
[Ni(L)Cl ₂]	(14.05)	(54.58)	(3.83)	(6.70)	(16.99)
	13.86	53.96	3.28	6.04	16.42
[Cu(L)Cl ₂]	(15.03)	(53.96)	(3.78)	(6.62)	(16.80)
	14.86	53.74	3.33	6.15	16.24
[Cd(L)Cl ₂]	(23.84)	(48.36)	(3.39)	(5.94)	(15.06)
	23.64	48.02	2.84	5.32	14.86

Table-3: Importante bands (cm^{-1}) of infrared spectra for free ligand and it's complexes

Compound	VC=N	VC-N	VM-N
Ligand (L)	1612 (s)	1010 (s)	----
C ₁₉ H ₁₆ N ₂ O	1550 (w)		
[Mn(L)Cl ₂]	1594 (s)	1016 (m)	590 (w)
[Fe(L)Cl ₂]	1594 (s)	1016 (s)	594 (w)
[Co(L)Cl ₂]	1596 (m)	1016 (s)	586 (w)
[Ni(L)Cl ₂]	1592 (m)	1014 (m)	588 (w)
[Cu(L)Cl ₂]	1598 (s)	1016 (s)	592 (m)
[Cd(L)Cl ₂]	1596 (m)	1016 (m)	586 (m)

S=strong, m=medium, w=weak

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Table-4: Magnetic moment (μ_{eff}) and U.V-Vis spectral data in DMSO for free ligand and it's complexes

Compound	Meff	λ	ν^-	ϵ_{max}	Assignment	Suggested Structure
	B.M	nm	cm ⁻¹	L.mol ⁻¹ .cm ⁻¹		
Ligand (L)	---	278	35971	2148	$\pi \rightarrow \pi^*$	---
C ₁₉ H ₁₆ N ₃ O		299	33445	235	$n \rightarrow \pi^*$	
[Mn(L)Cl ₂]	5.42	272	36764	2142	$\pi \rightarrow \pi^*$	Tetrahedral
		302	33112	2348	$n \rightarrow \pi^*$	
		382	26178	286	C.T	
		685	14598	98	${}^6A_1 \rightarrow {}^4T_1$	
[Fe(L)Cl ₂]	4.35	270	37037	2142	$\pi \rightarrow \pi^*$	Tetrahedral
		300	33333	2354	$n \rightarrow \pi^*$	
		380	26315	290	C.T	
		788	12690	11	${}^5E \rightarrow {}^5T_2$	
[Co(L)Cl ₂]	4.62	265	37736	2146	$\pi \rightarrow \pi^*$	Tetrahedral
		299	33445	2358	$n \rightarrow \pi^*$	
		396	25253	270	C.T	
		676	14793	193	${}^4A_2(F) \rightarrow {}^4T_1(P)$	
[Ni(L)Cl ₂]	3.81	272	36764	2144	$\pi \rightarrow \pi^*$	Tetrahedral
		302	33112	2348	$n \rightarrow \pi^*$	
		382	26178	286	C.T	
		525	19047	18	${}^3T_1(F) \rightarrow {}^3T_2(F)$	
		680	14706	17	${}^3T_1(F) \rightarrow {}^3T_1(P)$	
[Cu(L)Cl ₂]	1.94	270	37037	2146	$\pi \rightarrow \pi^*$	Tetrahedral
		300	33333	2352	$n \rightarrow \pi^*$	
		392	25516	192	C.T	
		776	12886	41	${}^2T_2 \rightarrow {}^2E$	
[Cd(L)Cl ₂]	Diamagnetic	270	37037	2146	$\pi \rightarrow \pi^*$	Tetrahedral
		301	33223	2352	$n \rightarrow \pi^*$	
		380	26315	200	C.T	

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Table-5: 1H NMR spectral data for free ligand(L) measured in DMSO- d_6

Functional group	δ (ppm)
-CH ₃	1.34(3H,S)
DMSO	2.78(S)
(Ar-CH)five ring	5.75(H,S)
	6.40(2H,S)
Ar-CH	7.32(4H,S)
Ar-CH	7.60(4H,S)
-N=CH	8.10,8.48(2H,S)

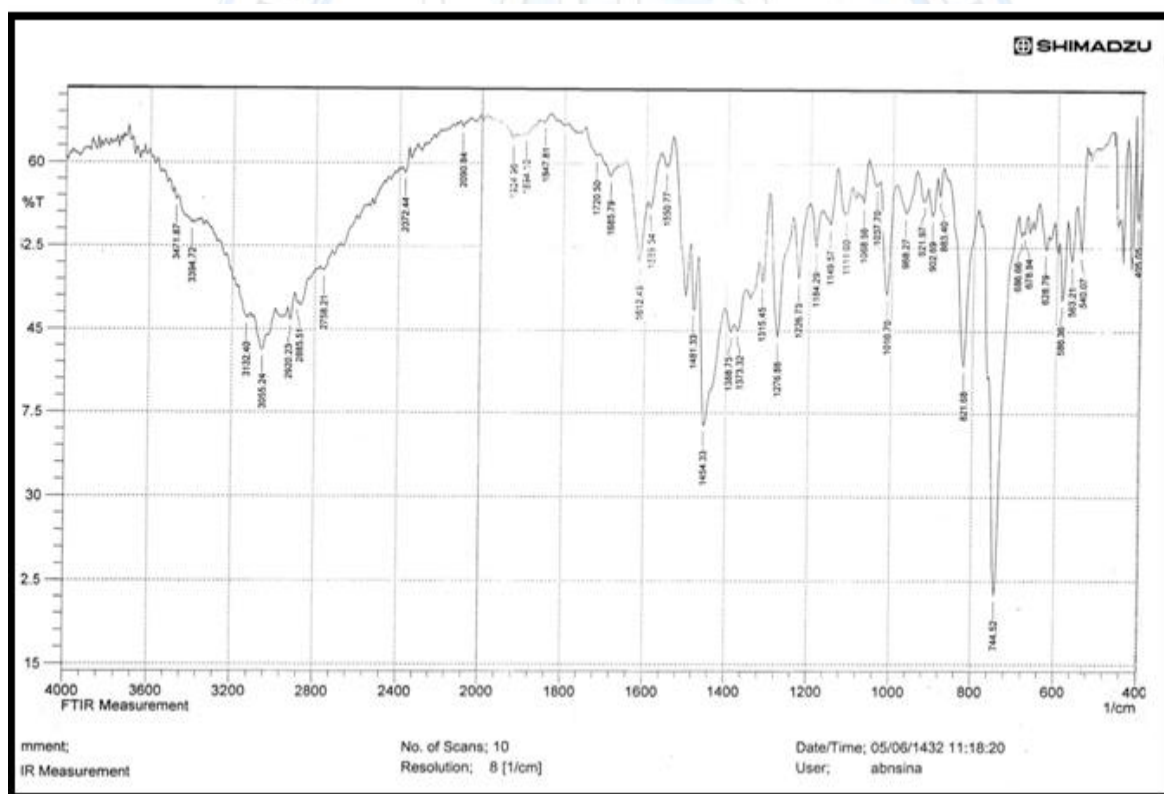


Fig.1: IR spectrum of ligand (L)

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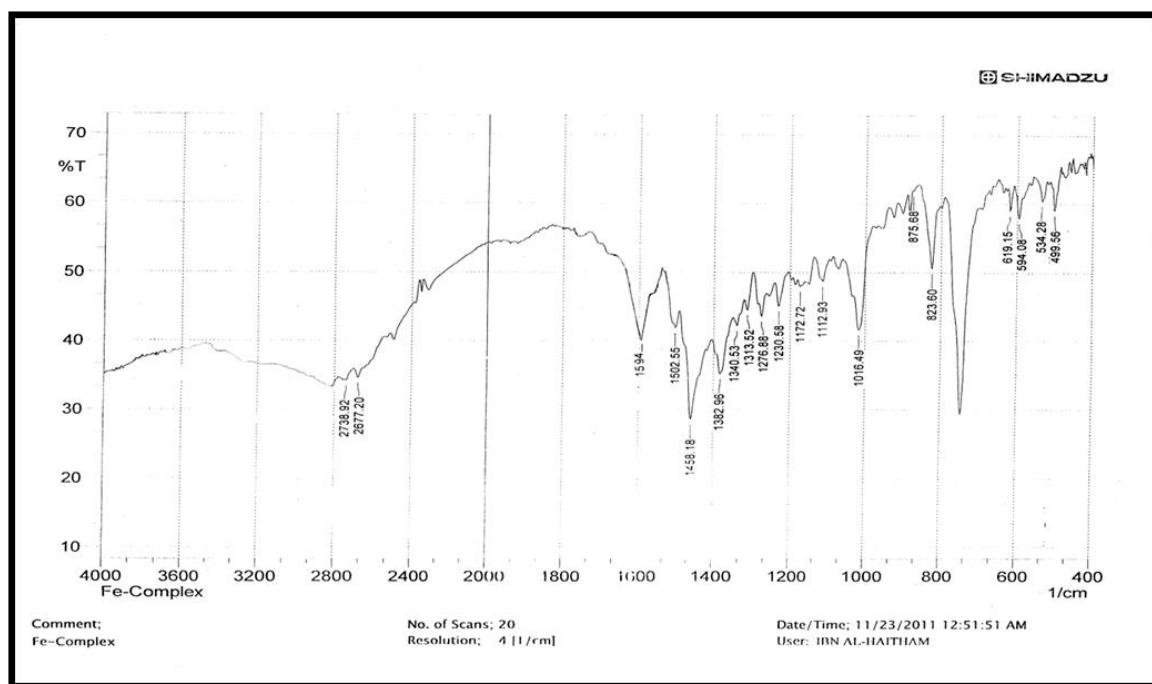


Fig.2: IR spectrum of Fe(II) complex

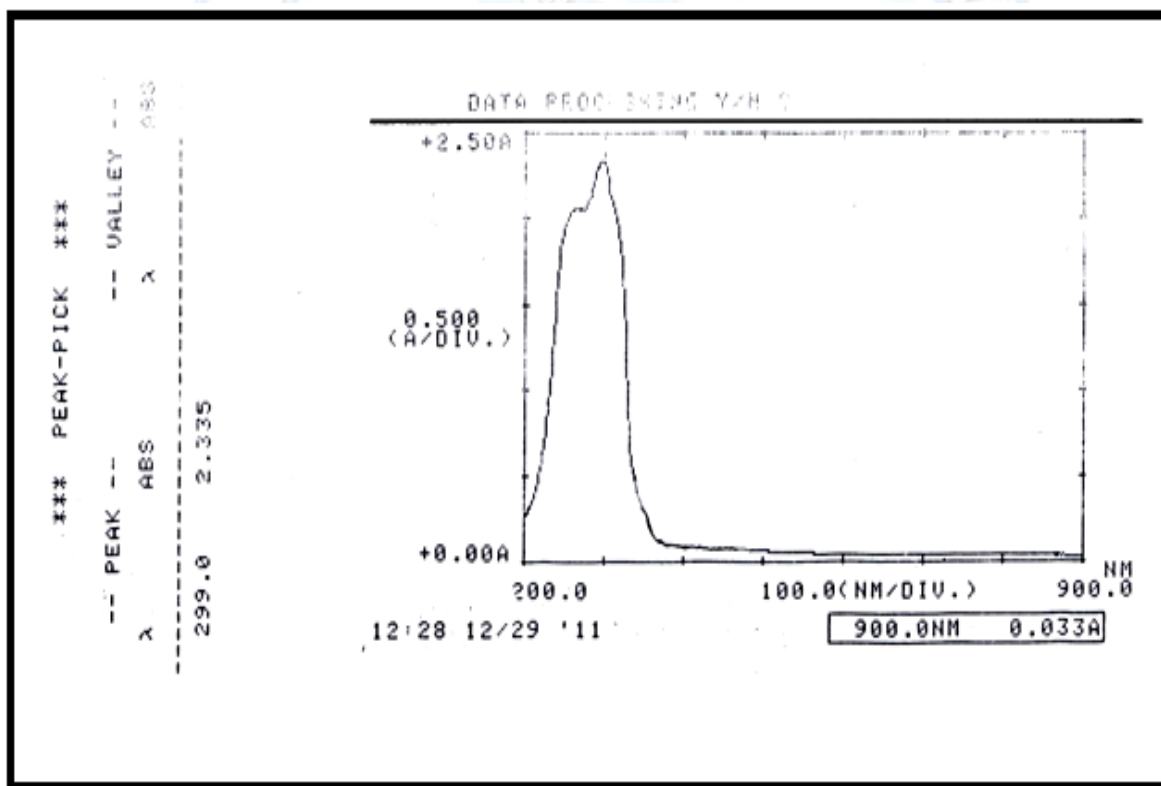


Fig.3: Electronic spectrum of ligand (L)

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Waleed K.M, *Haider D.J, Kwalha M.S and Sajid M.L ,

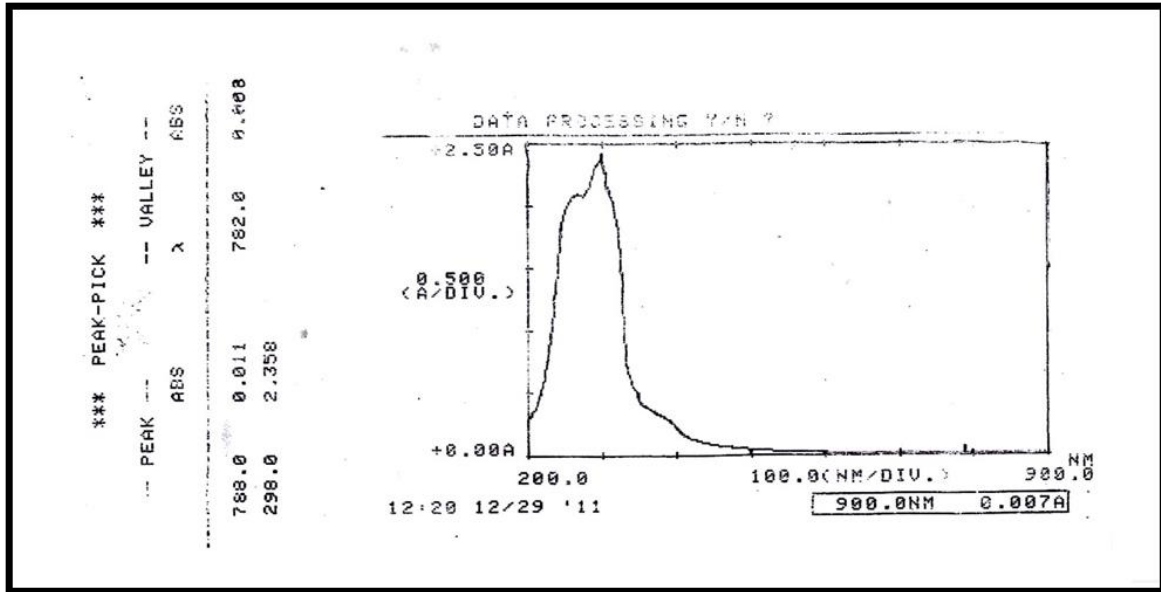


Fig-4: Electronic spectrum of Fe(II) complex

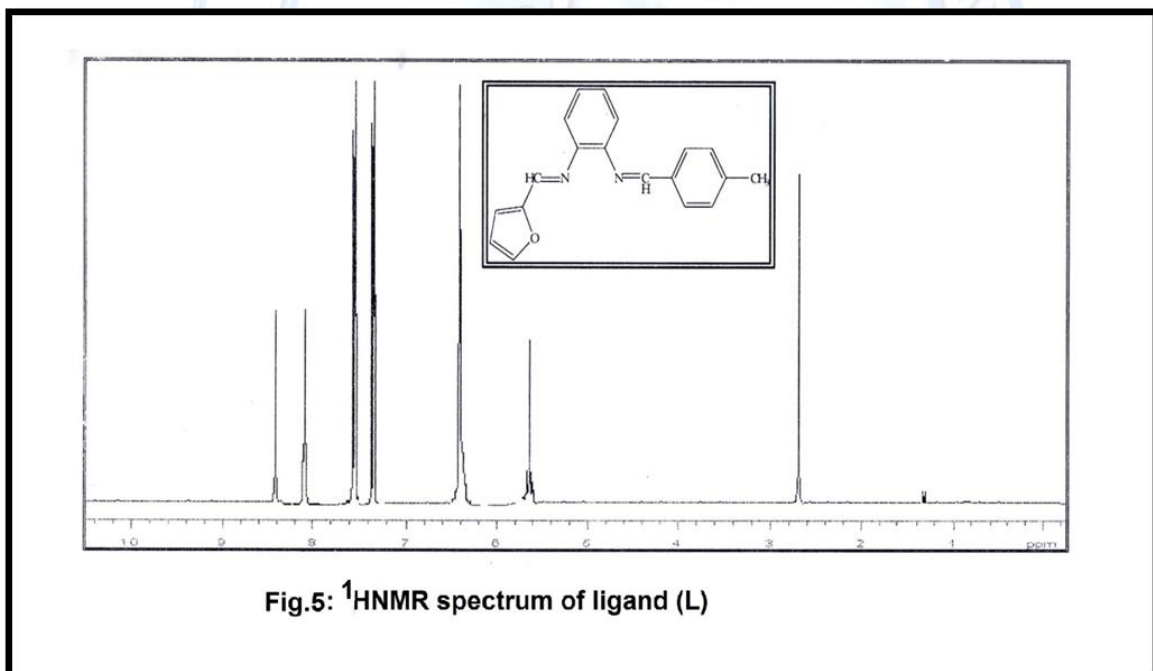


Fig.5: 1H NMR spectrum of ligand (L)