

Synthesis and Characterization of New Schiff Base Ligand Derived from
4 –aminoantipyrine and it's complexes with some Metal Ions and
Their Antibacterial Study

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Abstract

Metal complexes of Schiff base ligand(L), prepared via condensation of 4–aminoantipyrine and benzil, are prepared. The ligand is characterized based on elemental analysis , IR, electronic spectrum , ¹HNMR along with melting point. Metal complexes are reported and characterized based on elemental analysis , IR , electronic spectra , magnetic moment , molar conductance , ¹HNMR (for zinc complex only) and chloride content along with mole ratio method and melting point . From the elemental analyses and mole ratio method , 1 : 2 metal : ligand for Mn(II) and Cu(II) complexes and 1:1 metal : ligand for Zn(II) and Cd(II) complexes, are found with molecular formula : [Mn (L)₂Cl₂]. H₂O, [Cu(L)₂(H₂O)Cl]Cl, [Zn(L)Cl₂]. H₂O and [Cd (L)Cl₂] .

From the IR results , we suggested that the ligand (L) behaves as bidentate on complexation with metal ions via the azomethine nitrogen and carbonyl oxygen atom of five member ring of the ligand .The electronic spectral data and magnetic measurements indicate that the complexes exhibit octahedral geometry around Mn(II) and Cu(II) while tetrahedral geometry around Zn(II) and Cd(II) . The results of antibacterial activity showed that only Cd(II) complex have a high activity (22mm) for Eschirichia coli .

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Key words : 4- aminoantipyrine , Schiff base , Benzil , antibacterial activity , Zinc complex .

تحضير وتشخيص ليكاند قاعدة شف جديدة مشتقة من 4 – أمينو أنتي بايرين ومعقداتها مع بعض
الأيونات الفلزية ودراسة فعاليتها المضادة للبكتيريا

ساجد محمود لطيف و هيام هادي علكم

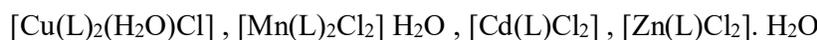
جامعة بغداد / كلية التربية للعلوم الصرفة / ابن الهيثم – قسم الكيمياء

العراق – بغداد – الاعظمية

المخلص

تم تحضير معقدات ليكاند قاعدة شف جديدة مشتقة من تكثيف 4- أمينو أنتي بايرين والبنزينيل. شخص الليكاند بالتحليل الكمي الدقيق للعناصر، طيف الأشعة تحت الحمراء ، طيف الأشعة فوق البنفسجية – المرئية ، بروتون الرنين النووي المغناطيسي فضلاً عن قياس نقطة الانصهار. اما المعقدات الفلزية المحضرة فقد شخصت أيضاً بالتحليل الكمي الدقيق للعناصر، وأطياف الأشعة تحت الحمراء ، الأطياف الالكترونية ، طيف بروتون الرنين النووي المغناطيسي (لمعقد الزنك فقط) ، قياس نقطة الانصهار فضلاً عن العزم المغناطيسي، إيجاد محتوى الكلور، قياس التوصيلية المولارية وإيجاد النسبة المولية فلز : ليكاند . من معطيات التحليل الكمي الدقيق للعناصر وطريقة النسب المولية وجد أن النسبة المولية فلز : ليكاند ، 1 : 2 لمعقدي المنغنيز (II) والنحاس (II) بينما

تساوي 1 : 1 لمعقدي الزنك (II) والكاديوم (II) وبصيغ جزيئية هي



نتائج طيف الأشعة تحت الحمراء أثبت أن الليكاند يسلك بصفته ثنائي السن عن طريق نتروجين الازوميثين وأوكسجين الكاربونيل للحلقة الخماسية . اما معطيات الأطياف الالكترونية والعزوم المغناطيسية للمعقدات فقد اشرت أن لمعقدي Mn ، Cu شكل ثماني السطوح بينما يكون الشكل رباعي السطوح لمعقدي Cd, Zn .

نتائج دراسة الفعالية المضادة للبكتيريا بيّنت أن معقد Cd فقط يمتلك فعالية عالية (22mm) مضاد لبكتيريا Escherichria Coli .

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الكلمات المفتاحية: 4-امينو انتي بايرين ,قاعدة شف , بنزيل , فعالية مضادة للبكتيريا ,معدن الزنك .

Introduction

Schiff bases and their metal complexes are of growing importance in coordination chemistry, attributable to recent observation in antibacterial antifungal and oxygen carrier properties^[1-3].

The investigations of structure and bonding of Schiff bases complexes help understand the complexes. Most of the biological systems possesses metal ions in unsymmetrical environments .

These structures can be modified through condensation with aldehyds and ketones^[4-6]. Schiff bases derived from 4 – aminoantipyrine and their transition metal complexes have been extensively examined due to their wide applications in various fields like biological , pharmacological , clinical, analytical and industrial area^[7-9]. Schiff bases of 4 – aminoantipyrine have some antimicrobial activity which have been enhanced by chelating it with transition ions, such as their complexes with Pt (IV) and Co(II)^[10,11].

A search through the literature reveals that no work has been done on the transition metal complexes of Schiff base formed by the condensation of 4 – aminoantipyrine with benzil.

In the present context , we synthesized bidentateligand system from 4 – aminoantipyrine and benzil. The synthesized compound and its chelation behavior with metal ions was studied ,

Experimental

Chemicals

Regent grade 4 – aminoantipyrine , benzil were obtained from Fluka and used as received while metal salt (Manganese chloride tetrahydrate , copperchloride dihydrate , zinc chloride and cadmium chloride monohydrate) were available from, B.D.H chemicals . The solvents and other chemicals were used without purification .

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Instrumentation

The IR spectra for prepared compounds were recorded as KBr discs using a shimadzu 8300S FTIR spectrophotometer in range (4000 – 400) cm^{-1} . Electronic spectra were measured in region (200– 900)nm for 10^{-3}M solution in DMSO at 25°C using shimadzu160 A spectrophotometer , with 1.000 ± 0.001 cm matched quartz cell. Elemental microanalysis were performed on a (C.H.N) analyzer from (Elemental microanalyses, Euro vector, model EA 3000 A origin: Italy). ^1H NMR spectrum of prepared ligand (L) and zinc complex were recorded on ultra shield 300 MHZ, Bruker, Switzerland , using DMSO – d^6 as a solvent .

The chloride content for complexes were determined by using potentiometric titration method on (686 – swiss), while metal contents of the complexes were determined by atomic absorption (A.A) technique using a shimadzu AA 680. Electrical conductivity measurements of the complexes were recorded at 25°C for 10^{-3}M solution of the sample in methanol using (BC 3020 professional Benchtop conductivity).

Magnetic susceptibilities were recorded on Bruker BM6 Instrument at 298°K following the Farady's method and using diamagnetic corrections were calculated with Pascal's constant . In addition melting points were obtained using (start melting point Apparatus) type Digimelt (MSRS).

The proposed molecular structure of the compounds were drawing by using chem. Office prog. 3DX (2006) .

Synthesis of Schiff base ligand

A solution of 4 – aminoantipyrine (0.203, 1mmol) in (25ml) ethanol was added to an ethanolic solution(20ml) of benzil (0.42g , 1mmol) with drops from glacial acetic acid^[12] .

The mixture was allowed to reflux for (8hrs) at 56°C then cooled at room temperature . A yellow solid was collected by filterition, washed with ethanol and recrystalized from methanol to give yellowish crystals (scheme-1) . Yield 81 % , m. p. ($186^\circ - 188^\circ\text{C}$) .

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Synthesis of Mn(II) , Cu(II) , Zn(II) and Cd(II) complexes

An ethanolic solution (25ml) of metal (II) chloride (1 mmol) was stirred with an ethanolic solution (25ml) of prepared Schiff base ligand (0.79 g , 2 mmol) and the resultant mixture was refluxed for (2hrs) .

Then the solution was reduced to one- third on water path. The solid complex precipitated was filtered off, washed with ethanol and dried under vacuum to give metal complex (scheme- 2) .

Antimicrobial

Antimicrobial activities of the ligand and the complexes have been carried out against the pathogenic bacteria like Escherichia coli and staphylococcus aureus using nutrient agar medium by disc diffusion method . The test solution were prepared in DMSO and soaked in filter paper of 5 mm diameter and 1 mm thickness . These discs were placed on the already seeded plates and incubated at 37°C for 24hrs . The diameters (mm) of the inhibition zone around each disc were measured after 24 hrs. streptomycin was used as standard^[13] .

Results and Discussion

The Schiff base ligand and its Mn(II), Cu(II), Zn(II) and Cd(II) complexes have been synthesized and characterized by spectral and elemental analytical data . They are found to be air stable. The ligand is soluble in common organic solvents and all complexes are freely soluble in CH₃OH , CCl₄ , CHCl₃, DMSO and DMF . The elemental analysis results for the ligand and their metal complexes are in a good agreement with the calculated values . (Table-1) shows the Stoichiometry of the metal (II) complexes. The molar conductance of 10⁻³M of the complexes in CH₃OH solvent at 25°C were measured (Table-3). The lower conductance values of Mn, Zn and Cd complexes support non- electrolytic nature while the conductance value for Cu complex indicated electrolytic nature with 1:1 ratio .

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¹HNMR spectra

The ¹HNMR spectra of the Schiff base ligand and its zinc complex were recorded at room temperature in DMSO – d⁶.

The aim of this work was to compare the chemical shifts of different protons for Schiff base ligand with that recorded for zinc complex. The predicted ¹HNMR spectrum of the free ligand (Fig.1) showed signals at: multiplet signals for – Ar – CH at range (7.51–7.71) ppm, singlet signal for – C – CH₃ at 2.47 ppm and doublet signals for – N – CH₃ at (3.13, 3.32) ppm.

However the ¹HNMR spectrum of the zinc complex (Fig.2) showed signals of coordinated ligand, shifted downfield and observed multiplet signals for – Ar – CH at range (6.91–7.71)ppm, multiplet signals for – C– CH₃ at range(1.87–2.51)ppm and doublet signals for– N – CH₃ at (2.89, 3.13)ppm. The splitting pattern of the zinc complex shows that rigidity is generated in the molecule as a result of coordination. In addition the ¹HNMR spectrum of zinc complex revealed the stability of it in DMSO solvent. [15, 16]

IR Spectra

The coordination mode and sites of the ligand to the metal ions were investigated by comparing the infrared spectra of the free ligand with its metal complexes (Table -2).

The band at 1660 cm⁻¹ in the spectrum of the free ligand (Fig.3) assigned to ν C = O of 4-aminoantipyrine based Schiff base ligand and, shifted towards lower values at range (1650 - 1656)cm⁻¹ indicating the coordination of oxygen atom of these carbonyl group^[15,17]. The spectrum of free ligand shows a band in the region 1562cm⁻¹ characteristics of the ν C=N(azomethine) stretching mode indicating the formation of Schiff base product. This band was shifted towards lower frequencies in the spectra of its complexes (1541 -1552)cm⁻¹ compared with the above Schiff base indicating the involvement of the azomethine nitrogen atom in the coordination with metal ion^[18,19]. The IR spectra of Mn(II) (fig.4) and Zn(II) complexes showed a broad band around (3383, 3566)cm⁻¹ respectively, which can be attributed to

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$\nu(\text{O-H})$ for lattice water^[20], while the IR spectrum of Cu(II) complex showed two bands at 316 cm^{-1} and 935 cm^{-1} which can be attributed to $\nu(\text{O-H})$ and $\delta(\text{O-H})$ for coordinated H_2O Molecules (aqua) with Cu(II) ion^[21]. Conclusive evidence of the bonding is also shown by the observation that new bands in the spectra of all metal complexes appearing in the low frequency regions 505 cm^{-1} and 437 cm^{-1} characteristic to $\nu(\text{M-N})$ and $\nu(\text{M-O})$ stretching vibration respectively, that are not observed in the spectrum of free ligand^[15, 17].

Electronic Spectra and Magnetic Moment

The electronic spectra of the Schiff base complexes (fig.5 , fig.6), exhibited three peaks around (279 -293) nm, (342 , 345) nm and (364 – 379) nm (Table -3) related to intra- ligand $\pi \rightarrow \pi^*$, $n \rightarrow \pi^*$ and charge transfer (C.T) transition respectively^[22, 23]. The electronic spectra confirm the stability of these complexes in methanol solvent and supported the stability of complexes in the NMR solution. The structures of complexes in solutions are in accord with their structures in solid state. The electronic spectrum of Mn(II) complex displayed two new absorption peak, the first peak at 400 nm and another peak at 435nm assigned to (d-d) electronic transition type ${}^6\text{A}_{1g} \rightarrow {}^4\text{T}_{2g}(\text{D})$ and ${}^6\text{A}_{1g} \rightarrow {}^4\text{T}_{2g}(\text{G})$ respectively, suggesting octahedral geometry about Mn(II)^[12,24].

The magnetic moment value (5.42)B.M of this complex is typical for octahedral structure^[25]. The spectrum of the Cu(II) complex displayed a broad peak at 792nm attributed to (d-d) electronic transition type ${}^2\text{B}_{1g} \rightarrow {}^2\text{A}_{2g}$ of distorted octahedral Cu(II) complex^[24,26].

The magnetic moment value (1.92)B.M of this complex as well as the other analytical data, are in a good agreement with distorted octahedral structure^[27].

The electronic spectra of Zn(II) and Cd(II) complexes show no absorption peaks at range (367-900)nm, this indicates no (d-d) electronic transition happened (d^{10} - system) in visible region. These complexes are diamagnetic as expected, that is a good result for Zn (II) and Cd(II) tetrahedral geometry around this ions^[28].

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Chloride content

The chloride content measurements for all complexes (Table-1) gave approximated values for theoretical values .

The composition of the complexes

The composition of the all complexes formed in solution have been established by mole ratio method ^[29] . In this case the results revel 1:2 metal: ligand for Mn(II) and Cu(II) , while 1:1 metal : ligand for Zn(II) and Cd(II) complexes (Fig.7) . A chosen plots of were represented in for all complexes . The formation constant K_f of the 1:2 metal ligand for Mn(II) , Cu(II) and 1:1 metal : ligand for Zn(II) , Cd(II) complexes were evaluated spectroscopically using the following equations^[30] .

$$\alpha = \frac{A_m - A_s}{A_m} \quad \text{and} \quad K_f = \frac{1 - \alpha}{4 \alpha^3 C^2} \quad (1:2) \text{ metal : ligand}$$

$$K_f = \frac{1 - \alpha}{\alpha^2 C} \quad \text{for (1:1) metal : ligand .}$$

Where : C = the concentration of the complex in mol/ L ; α = degree of dissociation ;
As = the absorption of solution containing a stoichiometric amount of ligand and metal ion ;
Am = the absorption of solution containing the same amount of metal ion and excess of ligand.

The As and Am were measured at (λ max) of solution. The values of (As , Am , α , K_f , log kf) were tabulated in (Table -4).The high values of K_f may reflect the high stability of the prepared complexes .

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Antibacterial activity:

Antibacterial activity of ligand and Mn,Cu ,Zn, and Cd complexes against ane strain of Gram (+Ve) bacteria (staphyococcusaureus) and Gram (-Ve) bacteria (Escherichia coli) were recorded. The results indicate only Cd (II) complex have a high activity (22mm) for Escherichia coli. This can be attributed to an Cd(II) complex solution have ability to solvated the fit layer of bacteria cell and destroyed it.

Nomenclature of prepared ligand an its complexes

Table -5 shows nomenclature (IUPAC) of prepared ligand (L) and its complexes.

Conclusion

In this paper we have reported the coordination chemistry of complexes derived Schiff base ligand obtained from the reaction of 4 – aminoantipyrine and benzil with metal ions such as Mn(II), Cu(II), Zn(II) and Cd(II). The structure of the ligand and its prepared complexes were confirmed by elemental analysis, IR, u.v –visible, ¹HNMR, magnetic moment, molar conductance and chloride content.

The Schiff base ligand coordinates through its azomethine nitrogen and oxygen of carbonyl group of five member ring, to the central metal ion, The Schiff base behaves as a bidentate ligand on complexation with metal ions forming octahedral geometry for Mn(II) and Cu(II) complexes while tetrahedral geometry for Zn(II) and Cd(II).

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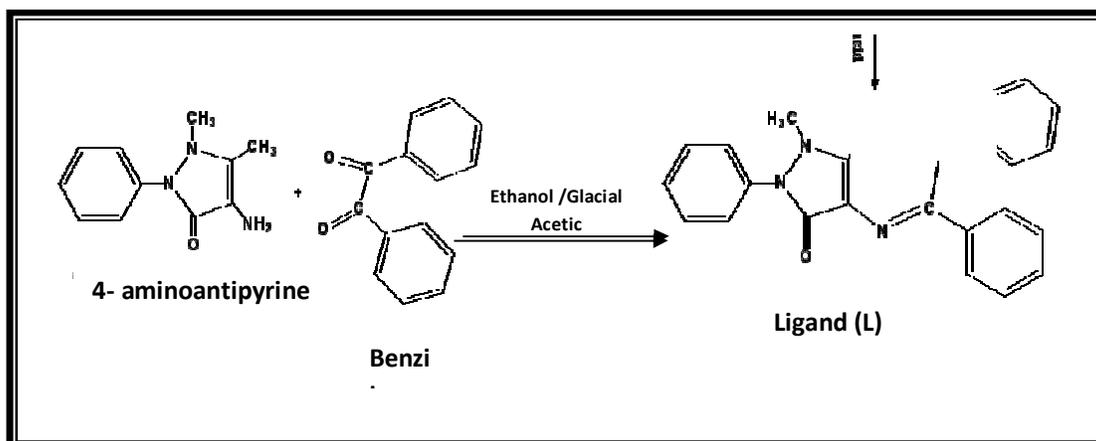
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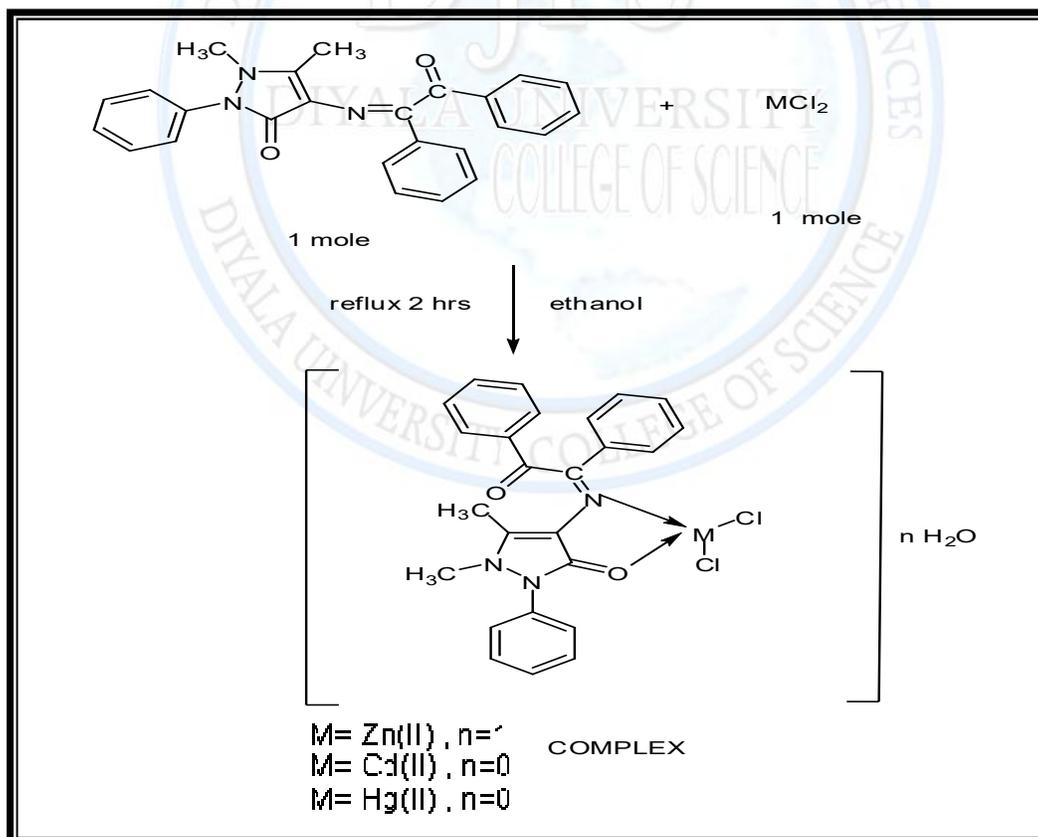
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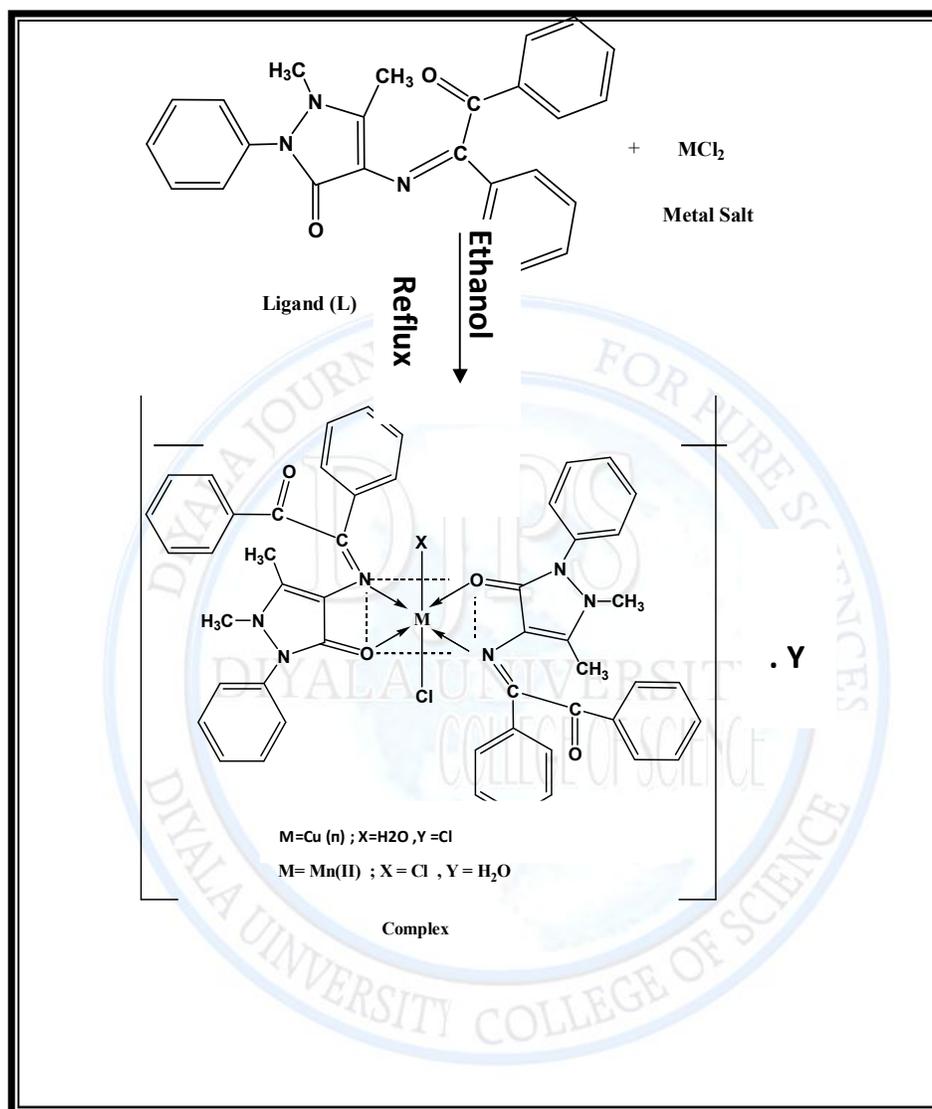
Scheme -1: synthesis route of the ligand (L)



Scheme -2: synthesis route of Zn(n) and Cd (n) Complexes

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Scheme-3 Synthesis route of Mn(n) and Cu(n) Complexes

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Table -1: Analytical and physical data of ligand (L) and its complexes

Compounds	M .Wt	Yield %	Colure	M.P. °C	Found % ,(calcu.%)				
					C	H	N	Metal	Cl
(L)=C ₂₅ H ₂₁ N ₃ O ₂	395	86	Yellow	186-188	75.42 (75.94)	5.48 (5.31)	11.02 10.63 (----	----
[Mn(L) ₂ Cl ₂].H ₂ O	934	71	Yellow ish white	168-170	62.94 (64.23)	3.26 (4.71)	10.08 (8.99)	5.24 (5.88)	6.84 (7.60)
[Cu(L) ₂ Cl(H ₂ O)] Cl	942.5	75	Reddish brown	98-100	62.92 (63.66)	5.24 (4.66)	7.38 (8.91)	6.15 (6.73)	8.41 (7.53)
[Zn(L) Cl ₂].H ₂ O	549.4	74	Yellow	147-149	54.28 (54.60)	5.06 (5.82)	6.85 (7.64)	10.97 (11.90)	11.92 (12.92)
[Cd(L)Cl ₂]	578.4	79	yellow	180-182	51.88 (51.86)	4.06 (3.63)	6.48 (7.26)	18.72 (19.34)	10.26 (12.27)

Table-2: Characteristic infrared absorption frequencies in (cm⁻¹) of ligand and its complexes

Compounds	VO-H	VC=O	vC=N (imine)	VC-N	VM-N	VM-O
(L) =C ₂₅ H ₂₁ N ₃ O ₂	-----	1660(vs)	1562(s)	1417(s)	-----	-----
[Mn(L) ₂ Cl ₂].H ₂ O	3383 (b)	1656(s)	1552(s)	1375(m)	505(w)	437(w)
[Cu(L) ₂ Cl(H ₂ O)]Cl	3169 (b)	1650(s)	1541(w)	1375(w)	505(w)	437(w)
[Zn(L) Cl ₂].H ₂ O	3566 (b)	1652(s)	1552 (s)	1375(m)	5050(w)	437(w)
[Cd(L)Cl ₂]	-----	1650(s)	1550(s)	1375(m)	505(w)	37(w)

b=broad s=strong w=weak m=medium

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**Table -3 :Electronic spectral data and conductance measurements of ligand(L)and its
complexes**

Compounds	$\lambda(\text{nm})$	$\nu(\text{cm}^{-1})$	ϵ_{max} $\text{L.mol}^{-1} .\text{cm}^{-1}$	Λ_{m} $\text{ohm}^{-1} .\text{cm}^2 .\text{mol}^{-1}$	Suggested structure
(L) =C ₂₅ H ₂₁ N ₃ O ₂	280 342 352	35714 29240 28409	1990 2032 1280	-----	-----
[Mn(L) ₂ Cl ₂].H ₂ O	279 342 364 400 435	35842 29240 25445 25000 22989	2016 1994 1240 500 300	10.18	Octahedral
[Cu(L) ₂ Cl(H ₂ O)]Cl	289 345 379 792	34602 28986 26385 12626	2177 1515 1201 31	43.41	Distorted octahedral
[Zn(L) Cl ₂].H ₂ O	279 342 364	35842 29240 25445	2018 1914 1220	3. 63	Tetrahedral
[Cd(L)Cl ₂]	293 342 366	34130 29240 27322	2159 2111 1304	5 .43	Tetrahedral

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**Table -4: Formation constant (K_f) of the prepared complex at $10^{-3}M$
and λ**

Compounds	As	Am	α	K_f	Log (K_f)
[Mn(L) ₂ Cl ₂]H ₂ O	2.388	2.506	0.047	0.229 x10 ¹⁰	9.36
[Cu(L) ₂ Cl(H ₂ O)]Cl	0.526	0.544	0.033	0.670 x10 ¹⁰	9.83
[Zn(L) Cl ₂]H ₂ O	1.524	1.601	0.048	0.413 x10 ¹⁰	5.616
[Cd(L)Cl ₂]	1.324	1.406	0.058	0.280x10 ¹⁰	5.448

Table -5: nomenclature of prepared ligand and its complexes

Compounds	Nomenclature
(L) C ₂₅ H ₂₁ N ₃ O ₂	1,5- dimethyl -4 - (2- oxo -1,2 - diphenylethylidene amino)-2 - phenyl -1,2 dihydropyrazol - 3- one
[Mn(L) ₂ Cl ₂] H ₂ O	dichlorobis[1,5- dimethyl -4 - (2- oxo -1,2 - diphenylethylidene amino)-2 - phenyl -1,2 dihydropyrazol - 3- one] manganese(II) Monohydrate
[Cu(L) ₂ Cl(H ₂ O)] Cl	aqua chlorobis [1,5- dimethyl -4 - (2- oxo -1,2 - diphenylethylidene amino)-2 - phenyl -1,2 dihydropyrazol - 3- one] Cupper(II) Chloride .
[Zn (L)Cl ₂] H ₂ O	dichloro[1,5- dimethyl -4 - (2- oxo -1,2 - diphenylethylidene amino)-2 - phenyl -1,2 dihydro pyrazol-3-one] Zince(II) Mono hydrate
[Cd (L)Cl ₂]	dichloro[1,5- dimethyl -4 - (2- oxo -1,2 - diphenylethylidene amino)-2 - phenyl -1,2 dihydropyrazol- 3- one]Cadimium (II) .

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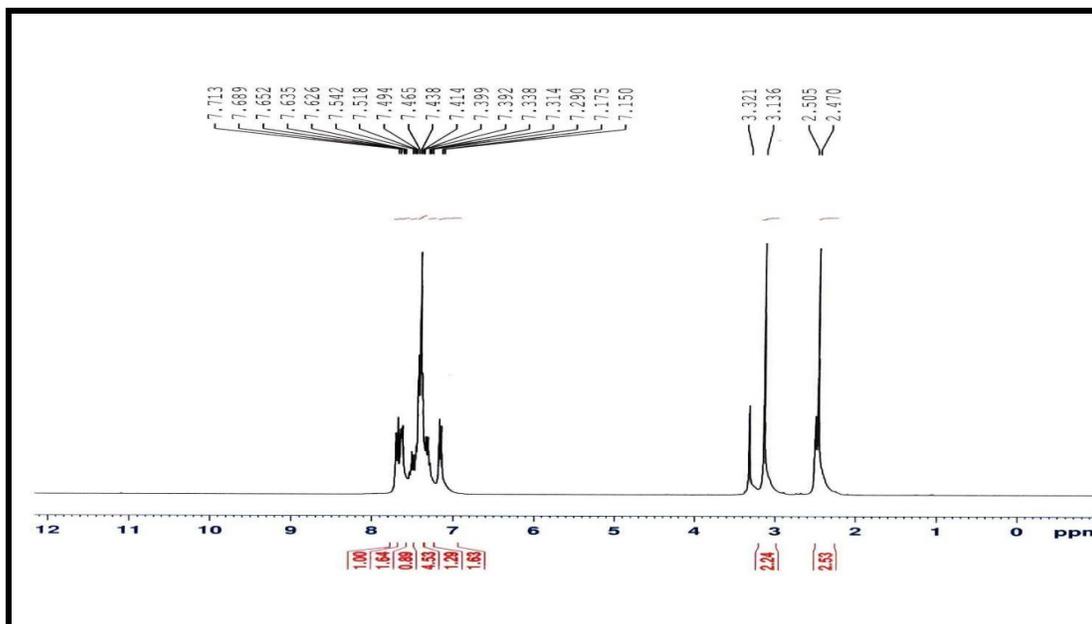


Fig .1: ¹H NMR Spectrum of Ligand (L)

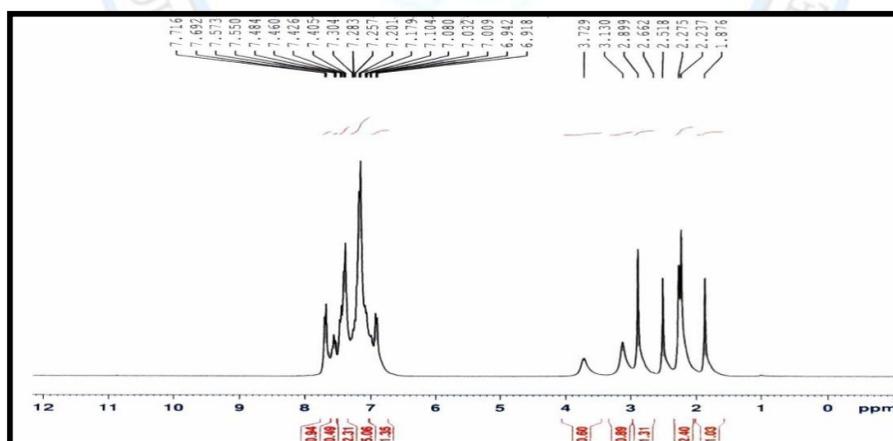


Fig .2: ¹H NMR Spectrum Of Zn (n) Complex

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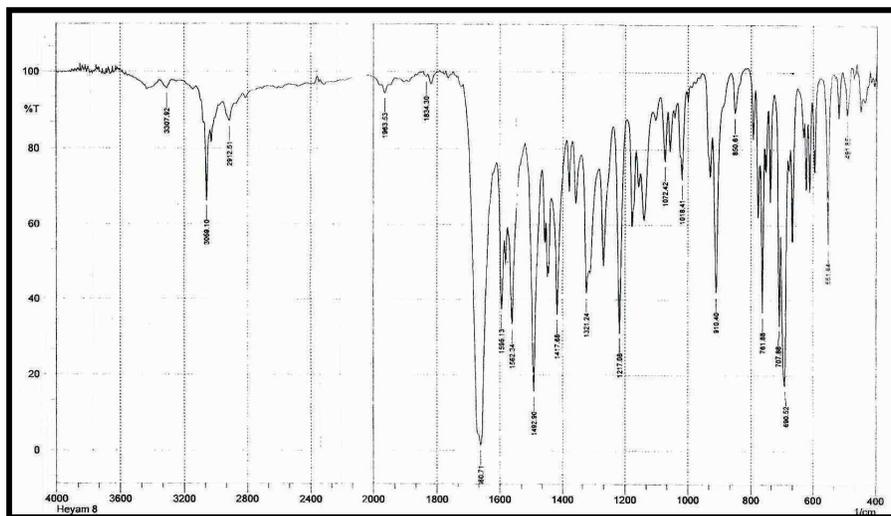


Fig .3: IR Spectrum of Ligand(L)

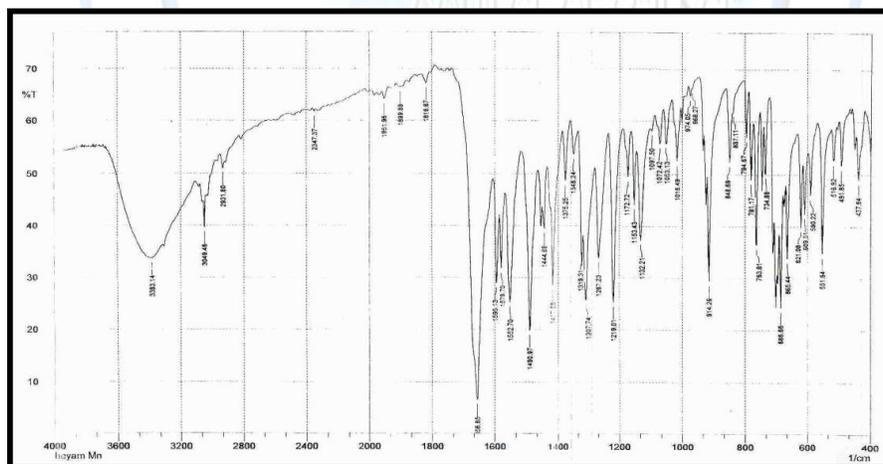


Fig .4: IR Spectrum of Mn(n)Complex

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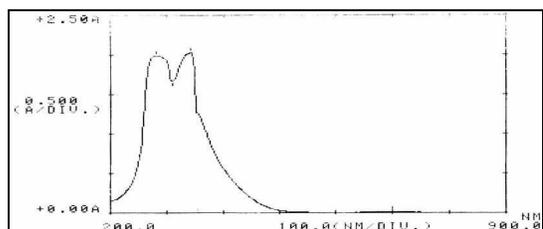


Fig.6:Electronic Spectrum of Cu (II) methodComplex

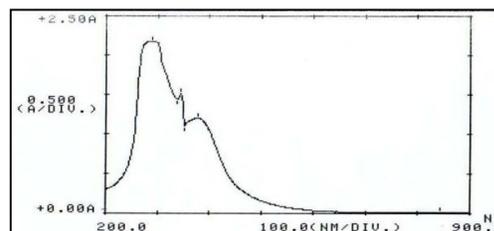


Fig .5: Electronic Spectrum of Ligand (L)

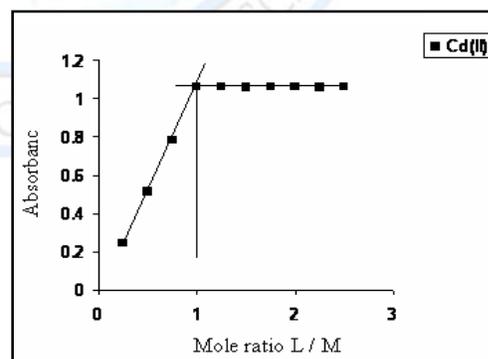
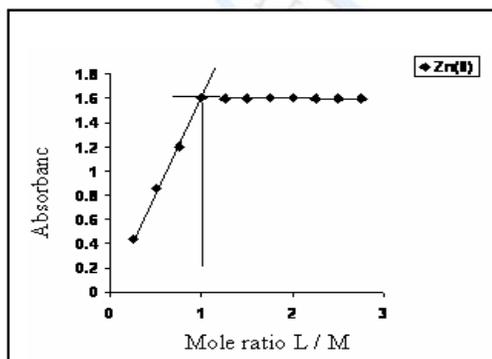
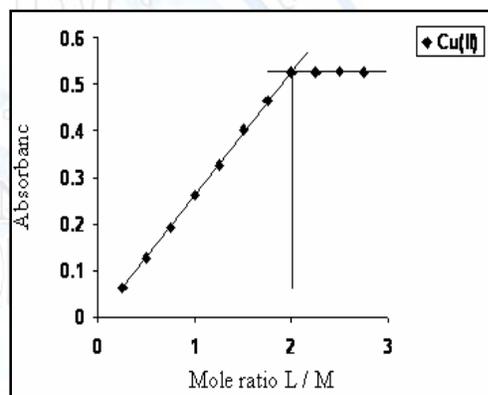
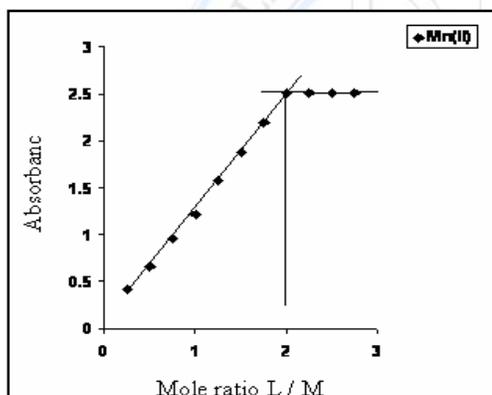


Fig .7: Mole ratio method for Mn(II) ,Cu(II) ,Zn(II) and Cd(II) Complexes