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Preparation and Spectroscopic Studies of New Transition Metal Complexes of 1-(2, 2-Dicyclohexylethyl)-3-(1, 5-Dimethyl-3-Oxo-2-Phenyl-2,3-Dihydro-1H-pyrazol-4-ylimino) Indolin-2-One

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Abstract

New tridentate ligand 1-(2, 2-dicyclohexylethyl)-3-(1, 5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-ylimino)indolin-2-one (L) was synthesized from the reaction of 3-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-ylimino)indolin-2-one and dicyclohexyl amine. Monomeric complexes of this ligand, of general formulae $[VO^{II}(L)(SO_4)]$, $[M^{III}(L)Cl_3]$ with M=Cr^{III} and Fe^{III}, and $[M^{II}(L)Cl]Cl$with (M = Mn^{II} and Co^{II}) are reported. The mode of bonding and overall geometry of the complexes were determined through FT-IR, UV–Vis, and mass spectral studies, magnetic moment measurements, elemental analysis, metal content, chloride containing and molar conductance. These studies revealed octahedral geometries for the Fe^{III}, Cr^{III} complexes, square pyramidal for $VO^{(II)}$ complex and tetrahedral for the Mn^{II} and Co^{II} complexes. The study of complexes formation via molar ratio and job method in DMF solution has been investigated and results were consistent to those found in the solid complexes with a ratio of (M:L) as (1:1). Hyper Chem-8 program has been used to predict structural geometries of compounds in gas phase the heat of formation (ΔH_f °), binding energy (ΔE_b), total energy, electronic energy and dipole moment at 298°k.

Keywords: Tridentate ligand, dicyclohexyl amine and mannch Schiff base complexes.

Introduction

Schiff bases compounds containing azomethine group (-C=N-) formed by condensation of primary amine and carbonyl compounds under acid or base catalysis or with heat [1], when aliphatic aldehydes are reaction relatively unstable and readily polymerizable while those of aromatic aldehydes, having an effective conjugation system are more stable [2]. Metal complex with Schiff bases are important class of ligands due to their synthetic flexibility, selectivity and sensitivity towards the central metal atom, structural similarities with natural biological substances, and also, due to presence of azomethine group which imports in elucidating the mechanism of transformation and racemisation reaction in biological system [3] also have been studied for their application in clinical, analytical and pharmacological areas [4]. Isatin as known (1H-indole-2,3-dione) possess both amide and keto carbonyl. The C-3 carbonyl group of isatin is strongly electrophilic and it readily undergoes condensation and addition reaction [5]. And through NH group compounds of the isatin series are capable of entering into N-alkylation,N-acylation and into the Mannich and Michael reaction [6]. Isatin Schiff bases are significant in therapeutic and pharmaceutical compounds in the field [7].

Experimental

The reagents were commercially available and used without further purification. Solvents were distilled from appropriate drying agents immediately prior to use. Elemental analyses (C, H and N) were performed using a Perkin-Elmer CHN 2400 elemental analyzer. The content of metal ions was calculated gravimetrically as metal oxides. Molar conductance measurements of the ligand and its complexes with (10^{-3} M) in DMSO were carried out using Jenway 4010 conductivity meter. Magnetic measurements were carried out on a Sherwood Scientific magnetic balance using the Gouy method. Electron impact (70 eV) mass spectra were recorded on a Finnegan-MAT model 8430 LC–MS-DS spectrometer. The UV-Vis spectra were obtained in DMF solution (10^{-3} M) for the ligand and its metal complexes with a Jenway

6405 spectrophotometer using 1 cm quartz cell, in the range 200–900 nm. IR spectra (4000–400 cm^{-1}) were recorded as KBr pellets on a Bruker FT-IR spectrophotometer.

Preparation of ligand: 1- (2, 2-dicyclo hexylethyl)-3-(1,5-dimethyl-3-oxo-2-phenyl - 2,3-dihydro-1H-pyrazol-4-ylimino) indolin-2-one

a- Preparation of 3-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-ylimino) indolin-2-one.

Equimolar (0.01 mol) quantity of indoline-2,3-dione and 4-Aminoantipyrine were dissolved in sufficient amount of ethanol and refluxed for 3 hrs in presence of glacial acetic acid. After standing for approximately 24 hrs. at room temperature, the products were separated byfiltration, dried under vacuum and recrystallized from war methanol.

b- Preparation of 1-(2,2-dicyclohexyl ethyl)-3-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-ylimino)indolin-2-one.

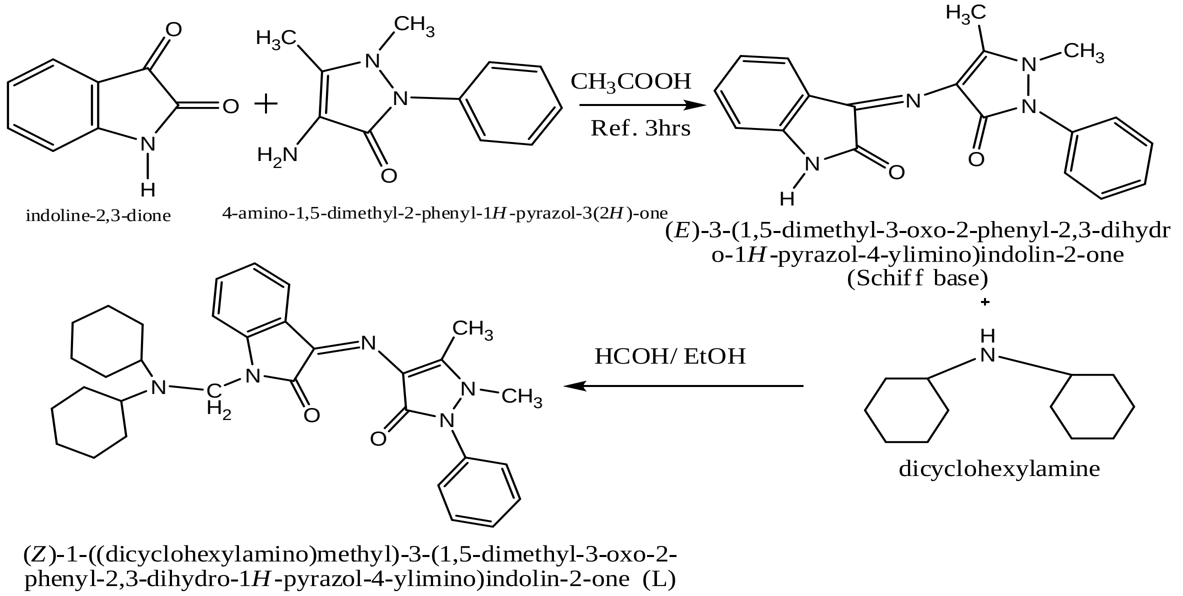
Equimolar quantity of dicyclohexyl amine (0.01mol) in 25 ml of ethanol was added to the solution containing Schiff bases and formaldehyde (37% v/v). The reaction mixture was stirred for 2 hrs atroom temperature and heating for 3 hrs followed kept under refrigeration for 24 hrs. The products were separated by suction filtration, dried under vacuum and recrystallized from ethanol. The molecular formula, molecularweight, melting point, yield and elemental analysis are shown in Table (1).

Preparation of Complexes

The preparation of all complexes is essentially the same and so a generic description will be presented. To a solution of (L) (1mmole) in ethanol was added slowly to a solution of metal salt ($VOSO_4$**.** H_2O, $CrCl_3$**.** $6H_2O$, $MnCl_2$. $4H_2O$, $FeCl_3.6H_2O$, and $CoCl_2.6H_2O$) in (ethanol and water) ratio (1:1) with stirring the mixture was refluxed for (4hrs). The solid was collected by filtration, recrystallized from methanol and dried at room temperature. Elemental analysis data, color and yield for the complexes are given in Table (1).

Results and discussion

The new (NO) and (ONO) dentate ligand L was obtained in good yield by the reaction of 3-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-ylimino)indolin-2-one and dicyclo hexylamine, Scheme 1. In general the ligand was characterized by elemental analysis Table (1), IR Table (2), UV-Vis Table (3), mass spectroscopy. Monomeric complexes of the ligand with Mn^{II}, Co^{II}, VO ^{II}, Fe^{III} and Cr^{III} were synthesized by heating (1 mmol) 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 $[VO^{II}(L)(SO_4)]$, $[M^{III}(L)Cl_3]$ with M^{III}=Cr and Fe, and $[M^{II}(L)Cl]Cl$ (M^{II} = Mn and Co) are reported, scheme (2). The complexes are air-stable solids, soluble in DMF and DMSO, sparingly soluble in MeOH, $CHCl_3$, CH_2Cl_2 and not soluble in other common organic solvents. The analytical data Tables (1) agree well with the suggested formulae.



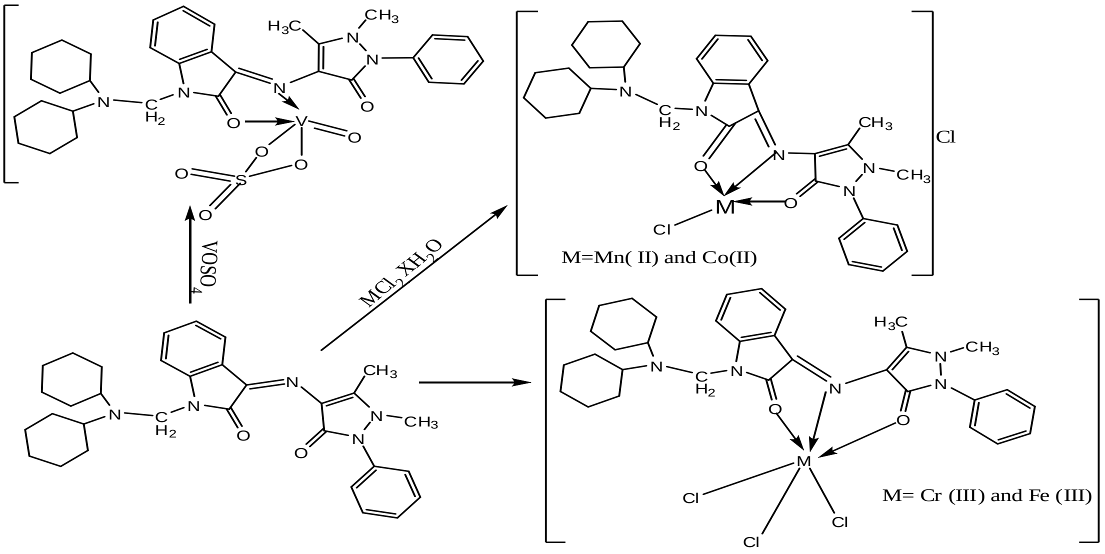
Scheme (1) Synthesis route of the ligand.

Table (1)

Analytical and physical data of the ligand and its complexes.

Compounds	***Formula M. wt***	***Color***	***M. P. ºC***	***Yield %***	***Elemental analysis, found (Calc.)%***				
					C	***H***	***N***	***M***	***Cl***
L	$C_{32}H_{39}N_5O_2$ 525.3	Orange red	156-158	87	73.16 (74.16)	7.42 (7.17)	13.32 (12.40)		
CoL	$C_{32}H_{39}N_5O_2Cl_2Co$ 655.13	Violate	265d	74	58.66 (60.06)	5.95 (5.53)	10.68 (10.04)	8.99 (10.00)	10.82 (10.98)
MnL	$C_{32}H_{39}N_5O_2Cl_2Mn$ 651.13	Yellow greenish	266d	75	59.02 (58.82)	5.98 (5.59)	10.75 (8.14)	8.43 (9.00)	10.88 (9.98)
FeL	$C_{32}H_{39}N_5O_2Cl_3Fe$ 687.64	Yellow greenish	222d	71	55.88 (58.82)	5.71 (5.59)	10.18 (8.84)	8.12 (9.00)	15.46 (9.98)
CrL	$C_{32}H_{39}N_5O_2Cl_3Cr$ 683.65	Yellow greenish	278d	82	56.21 (55.82)	5.70 (5.59)	10.24 (8.94)	7.60 (9.00)	15.55 (14.98)
VOL	$C_{32}H_{39}N_5O_7SV$ 688.31	Yellow greenish	280d	89	55.83 (56.82)	5.66 (5.59)	10.17 (8.94)	7.40 (9.00)	S= 4.66 (4.98)

d=decompose



Scheme (2) Proposed structures of metal complexes.

IR spectra

The IR spectra bands of the ligand (L) and its complexes were characterized at 3151-3089, 2966-2897, 1724-1651 and 1620 cm^{-1} due to the ν(CH) aromatic, ν(CH) aliphatic, ν(C=O), and ν(C=N) functional groups, respectively, for the ligand [9]. The IR spectraof the complexes exhibited ligand bands with the appropriate shifts due complexes formation [10]. Moreover, the ν(C=O), ν(C=N), bands of the ligand were observed at (1724,1651) cm^{-1}and 1620 cm^{-1} and these bands were shifted to the lower frequencies by (24-11, 27-11) and (42-9) cm^{-1} respectively in the spectra of the complexes. This indicates that the ligand was coordinated with the metal ions through the O, O carbonyl groups and N azomethine group atoms, but these shifts confirm the coordination of the ligand via the nitrogen of azomethine and the oxygen of carbonyl group to vanadyl ions. At lower frequency the complexes exhibited new bands around (588-508), and (450-413) cm^{-1} assigned to the υ(M-N)and υ(M-O), respectively [10,11].

Table (2)

FTIR spectral data (wave number υ^{-}) cm^{-1} for the ligand and its complexes.

Compound	*C-H arom*	*C-H alip*	*C=O*	*C=N*	*M-N*	*M-O*	
L	3151 3089	2966 2897	1724 1651	1620	-	-	
CoL	3099 3029	2956 2897	1700 1636	1578	570 508	440	
MnL	3111 3035	2939	1702 1627	1582	588	450 433	
FeL	3118 3065	2954 2864	1713 1624	1597	554	425 413	
CrL	3111 3078	2987 2894	1711 1640	1607	534	442	
VOL	3100 3042	2946 2853	1629	1611	555	432 449	υ(V=O)= 936 υ(SO_4)= 978, 951

arom = aromatic and alip = aliphatic

Electronic spectral, magnetic moments and conductivity measure ements

The electronic spectrum of the ligand exhibits intense absorption at (282,330) and 348 nm attributed to π→ π* and n→ π*, respectively. The electronic spectrum of Co(II) complex showed three broad peaks at 27397,15873 and 13986 cm^{-1} assigned to $^4A_{2\ F}\rightarrow {}^4T_{1(P)}$, $^4A_{2\ F}\rightarrow {}^4T_{1(F)}$ and $^4A_{2\ F}\rightarrow {}^4T_{2(F)}$ and the electronic spectrum of Mn(II) complex showed two broad peaks at 28490 and 27322 cm^{-1} assigned to $^6A_1\rightarrow {}^4E_{(D)}$ and $^6A_1\rightarrow {}^4T_{2(D)}$ respectively. The Fe$^{(III)}$ complex exhibited peak at 21739 cm^{-1} which assign to $^5T_2g \rightarrow {}^5Eg$ (D) transition, suggesting an octahedral geometry around the Fe$^{(III)}$ ion [13,14]. The spectra of the Cr$^{(III)}$ complex of together with the Mett value Table (3) suggest octahedral geometry around for complex. The spectrum of VO$^{(II)}$ complex gave one band at 15576 cm^{-1} assigned to $^2B_2 \rightarrow {}^2A_1$ and $^2B_2 \rightarrow {}^2E$ transition, respectively suggesting a square pyramidal geometry [15-17]. The magnetic moment value 3.54, 4.45 B.M. of Co(II), Mn(II) complexes respectively are typical for tetrahedral geometry [13]. The magnetic moment value at this complex consistent with octahedral geometry structure. The molar conductivity value of the complexes were consistent with non electrolytes for VO$^{(II)}$, Cr$^{(III)}$ and Fe$^{(III)}$ complexes and 1:1 electrolytes for Mn$^{(II)}$, Co$^{(II)}$ complexes. See the scheme (1) the preparation of the ligand and scheme (2) for its complexes.

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

Complexes	*Molar Conductive $cm^2\,s\,mole^{-1}$ in DMSO*	*μ_{eff} M. B.*	*λ_{max} nm*	*$\acute{\nu}$ cm^{-1}*	*ε max $Lmol^{-1}cm^{-1}$*	*Assignment*
CoL Tetrahedral	77	3.54	351 365 630 715	28490 27397 15873 13986	896 966 16 22	C. T $^4A_{2\,F}\rightarrow {}^4T_{1(P)}$ $^4A_{2\,F}\rightarrow {}^4T_{1(F)}$ $^4A_{2\,F}\rightarrow {}^4T_{2(F)}$
MnL Tetrahedral	59	4.45	296 345 351 366	33783 28985 28490 27322	1235 2500 882 946	(C.T) (C.T) $^6A_1\rightarrow {}^4E_{(D)}$ $^6A_1\rightarrow {}^4T_{2(D)}$
VOL Square Pyramidal	19	1.77	370 642	27027 15576	1157 43	C.T $^2B_2\rightarrow {}^2A_1$ $^2B_2\rightarrow {}^2E$
CrL Octahedral	88	3.13	357 425 510	28011 23529 19607	891 560 68	C.T $^4A_{2g(F)}\rightarrow T_{1P}$ $^4A_{2g(F)}\rightarrow T_{1F}$ $^4A_{2g(F)}\rightarrow T_{2F}$
FeL Octahedral	83	5.77	371 460	26954 21739	1171 187	C. T $^5T_2g\rightarrow {}^5Eg$ (D)

Mass spectrum

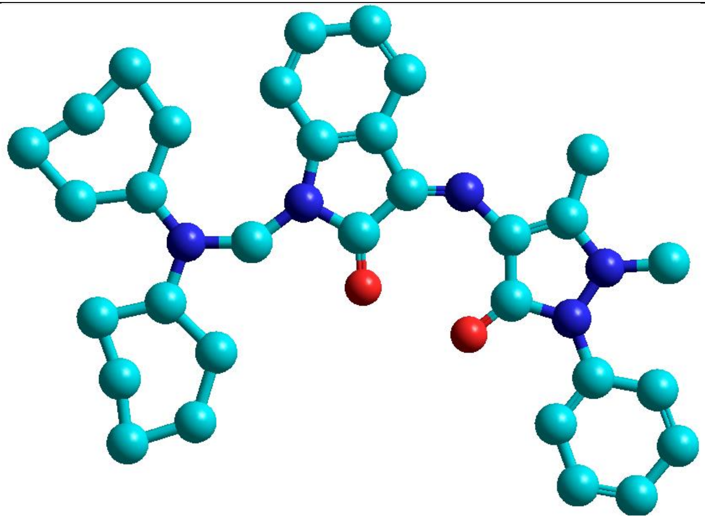
The electron impact spectrum of ligand (L) confirms the probable formula by showing a peak at (525.4 m/ z) cores bonding to mannch Schiff base moiety [($C_{32}H_{39}N_5O_2$)calculated atomic mass 525.4]. The series of peaks at 205 m/z is attributed to ($C_{11}H_{15}N_3O$), 204 m/z is attributed to ($C_{11}H_{14}N_3O$), 194 m/z is attributed to ($C_{13}H_{24}N$), 75 m/z is attributed to (C_3H_9NO) and 36 m/z is attributed to (H_6NO), and the fragmentation pattern for complexes confirms the probable formula by showing a peak at (655.4 m/ z) cores bonding to Co^{II} complex moiety [($C_{32}H_{39}N_5O_2CoCl_2$) calculated atomic mass 655.4]. The series of peak at 616 m/z is attributed to ($C_{32}H_{35}N_5O_2CoCl$), 430 m/z is attributed to ($C_{21}H_{17}N_5O_2Co$), 429 m/z is attributed to ($C_{21}H_{16}N_5O_2Co$), 205 m/z is attributed to ($C_{11}H_{15}N_3O$) and 204 m/z is attributed to ($C_{11}H_{14}N_3O$). And Mn^{II} complex moiety [($C_{32}H_{39}N_5O_2MnCl_2$) calculated atomic mass 651.5], the series of peaks at 614 m/z is attributed to ($C_{32}H_{37}N_5O_2MnCl$), 430 m/z is attributed to ($C_{18}H_{21}N_5O_2MnCl$), 429 m/z is attributed to ($C_{18}H_{20}N_5O_2ClMn$), 205 m/z is attributed to ($C_{11}H_{15}N_3O$) and 204 m/z is attributed to ($C_{11}H_{14}N_3O$).

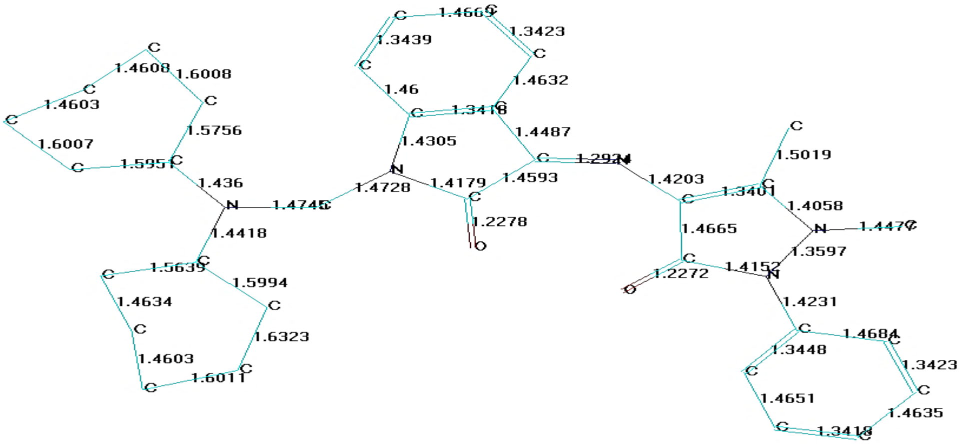
Theoretical study

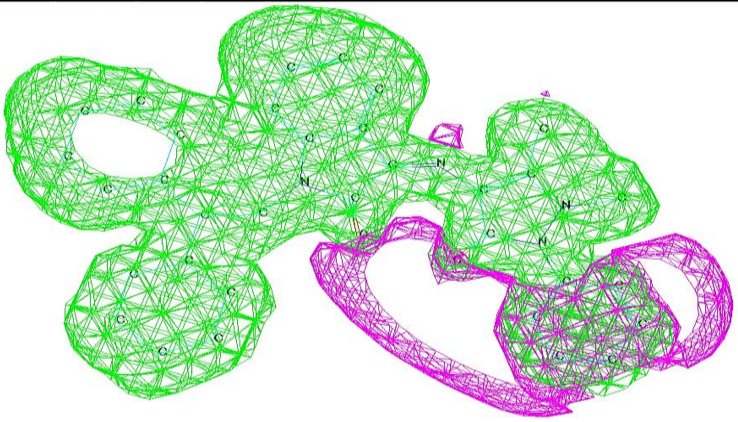
A theoretically probable structure of A metal complexes with ligand were calculated to search for the most probable model building stable structure, these shapes, show the calculated optima geometries for ligand. The results of PM3 method of calculation in gas phase for the binding energies and heat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

Table (4)
Conformation energetic in (K J.Mol^{-1}) for the ligand and complexes.

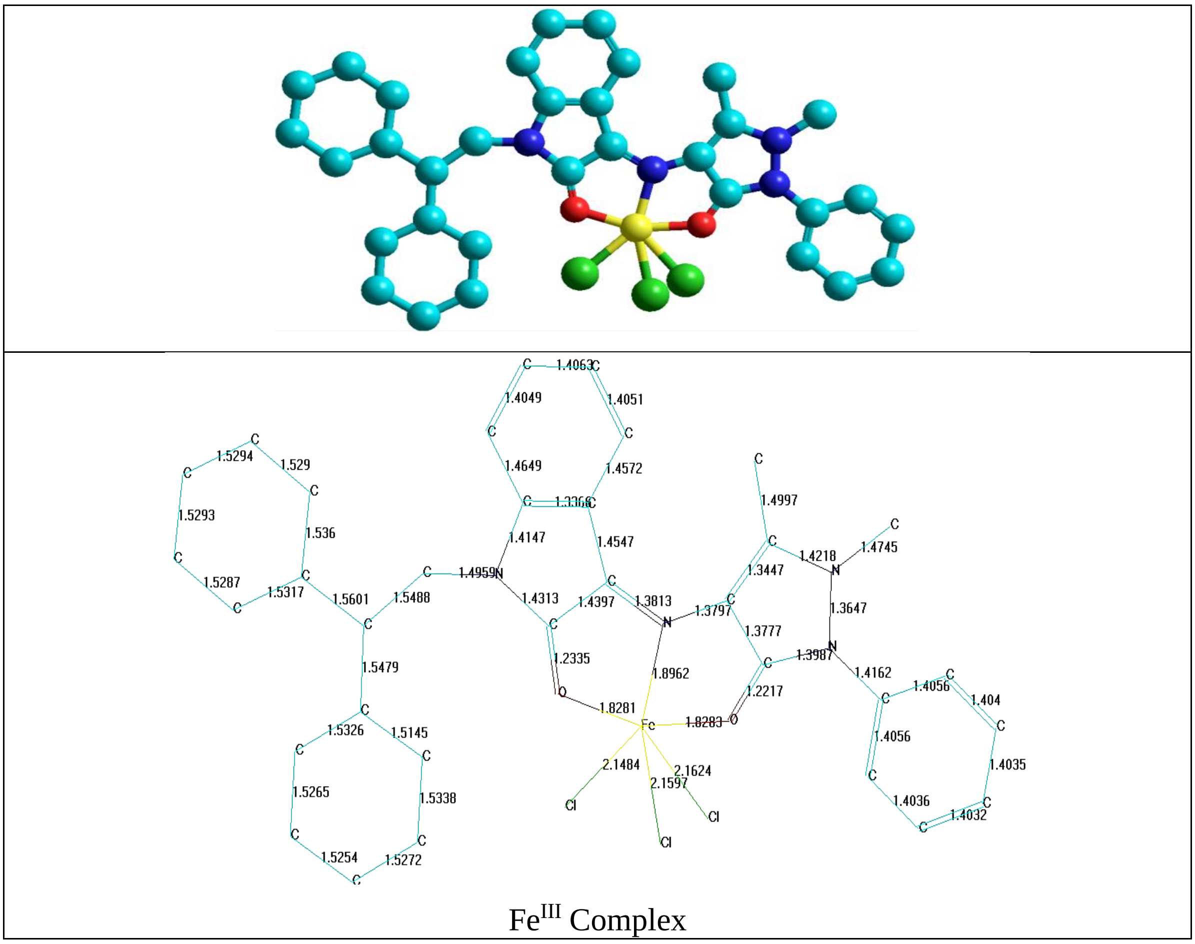
Compounds	*Binding Energy*	*Heat of Formation*	*Electronic Energy*	*Dipole moment (debyes)*	*Total Energy*
L	-4087.8472761	2122.6407239	-963880.021879	23.39	-116619.300806
CoL	-4326.4163316	2015.4616684	-1169716.1185115	19.648	-142058.723112
MnL	-4157.8280108	2149.3499892	-1135762.7660891	6.152	-132846.9020078
FeL	-4112.78009	1981.34446	-1117854.1178	8.156	-114221.76535
CrL	-3998.176532	1652.11655	-808754.11950	7.114	-219901.1675
VOL	-3398.1997601	1763.5667	-787154.458	9.875	-29998.11537







Electrostatic potential =-7.12203 eV for ligand



Fe^{III} Complex

Fig.(1): conformation structures, bond length of ligand and Fe^{III} complex.

Conclusion

In this paper synthesis and characterization of new mannich Schiff bases ligand 1-(2, 2-dicyclohexylethyl)-3-(1, 5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-ylimino)indolin-2-one (L) from reaction 3-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-ylimino) indolin-2-one (Schiff base) with dicyclohexyl amine and its preparation of complexes with the general formula [VO^{II}(L)(SO$_4$)], [M^{III}(L)Cl$_3$] with M=Cr^{III} and Fe^{III}, and [M^{II}(L)Cl]Cl with (M = Mn^{II} and Co^{II}) are reported. The molar conductivity of the complexes in DMSO solution was non-electrolyte for all complexes, but the Co^{II} and Mn^{II} complexes electrolyte (1:1), and the configurations were performed to coordinate the mannich Schiff base through the nitrogen and oxygen atoms. Therefore, from the presented results the complexes have tetrahedral for Co^{II} and Mn^{II} complexes , square pyramidal with VO^{II} complex and octahedral geometry with Fe^{III} and Cr^{III} complexes configuration. Theoretically probable structures of metal complexes with mannich Schiff base have been calculated, these shapes shows the calculated optima geometries for L and its metal complexes.

Acknowledgments

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الخلاصة

تم تحضير الليكاند الجديد 1-(2،2-ثنائي سايكلوهكسيل اثيل)-3-(1،5-ثنائي مثيل -3-اوكسو-2-فنيل-2،3-ثنائي هيدرو-1-بايروزول-4-ايل ايمينو) ايندولين-2-اون من تفاعل 3-(1،5-ثنائي مثيل-3-اوكسو-2-فنيل-2،3-ثنائي هيدرو-1-بايروزول-4-ايل ايمينو) ايندولين -2-اون مع ثنائي سايكلو هكسيل امين. استعمل اللكياند لتحضير المعقدات احادية الليكاند ذات الصيغ العامة $[VO^{II}(L)(SO_4)]$ و $[M^{III}(L)Cl_3]$ والـ M يمثل الكروم والحديد ثلاثي التكافؤ $[M^{II}(L)Cl]Cl$ والـ M يمثل الكوبلت والمنغنيز ثنائي التكافؤ. شخصت المركبات المحضرة لتحديد الترابط والاشكال الهندسية للمعقدات بتقنيات الاشعة تحت

الحمراء، الاشعة فوق البنفسجية –المرئية، طيف الكتلة، والتحليل الدقيق للعناصر، قياس محتوى الكلور، محتوى المعدن، التوصيلية المولارية الكهربائية والحساسية المغناطيسية. أظهرت الدراسة بهذه التقنيات الشكل ثماني السطوح لمعقدي الكروم والحديد ثلاثي التكافؤ ورباعي السطوح لكل من المنغنيز والكوبلت وهرم مربع القاعدة لمعقد الفناديل وان نسبة المعدن: الليكاند (1:1). درست المركبات المحضرة نظريا ببرنامج الهايبر كيم الثامن لتحديد حرارة التكوين وطاقة الترابط والطاقة الكلية وعزم ثنائي القطب بدرجة حرارة 298 كلفن وبالحالة الغازية لتحديد الاشكال الهندسية الاكثر استقراراً ثلاثية الابعاد واطوال الاواصر.