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Coordination Modes of a New Ligand Derived from Pyrazoline with Cr(III), Mn(II), Co(II), Ni(II), Cu(II) and Zn(II) Metal Ions; Synthesis, Identification and Biological Studies

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Abstract

The solid complexes of Cr(III), Mn(II), Co(II), Ni(II), Cu(II) and Zn(II) with 2-[5-(2-hydroxy phenyl) 1,3,4-oxadiazol-2yl]-5-methyl-2, 4 dihydro-3*H*-pyrazol-3-one] have been synthesized and characterized by elemental analysis, molar conductance, magnetic susceptibility, UV-Visible spectroscopy. The FT-IR data suggest that the ligand behaves as a neutral bidentate with NO donor atoms sequence towards central metal ion. The microanalytical data reveals the stereochemistry of the complexes has been found to be 1:1(metal:ligand) except that of Ni(II) complex which is formed in1:2 ratio. The physicochemical data suggest the octahedral geometry for all complexes except for Ni(II) and Zn(II) complex which were square planner and tetrahedral respectively.
The ligand (L) and its metal complexes were screened for their antibacterial activity against ***E-coli*** and ***Bascillus microorganisms*** andfungicidal activity against***Aspergillusnigar*** and ***Candida albicans*** with respect to standard antibiotic drugs tetracycline and Amphoteriain

Keywords: Pyrazol and Oxadiazol complexes.

1. Introduction

The coordination chemistry of transition metal complexes of heterocyclic compounds, involving oxadiazole, thiadiazoel and pyrazole ligand have attracted much attention in recent years due to the fact that those ligands around central metal ions in natural systems are unsymmetrical [1]ring closure reactions involvingoxadiazole and pyrozoles derivatives have carried out through hydrazides condensation with carbon disulfide in acidic medium[2-3]which are generally believed to show good models of biological systems [4]. The bidendate ligands of pyrazolines derivatives have been extensively examinal due to their wide applications in various fields like biological, analytical and therapeutical [5-6].Thus in this paper, we report the antifungal and antibacterial activites of new metal complexes of Cr(III), Mn(II), Co(II), Ni(II), Cu(II) and Zn(II) with the ligand and its metal complexes are characterized by physicochemical and spectra techniques.

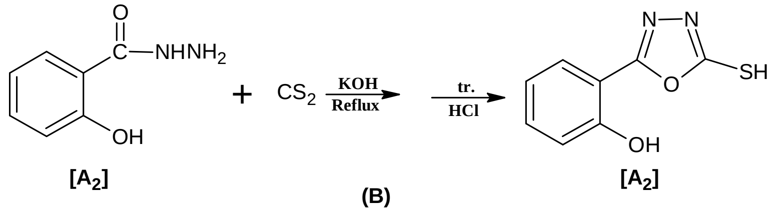
2. Experimental

2.1. Materials and Physical Measurements:

All starting materials and solvents were purchased from Aldrich and Fluka companies and used without further purification. Melting points of the prepared compounds were determined on electro-thermal capillary apparatus and are uncorrected, elemental analysis (C.H.N) were carried out using a Perkin-Elmer model 2400 instrument, FT-IRspectra were recorded on Shimadzu model FT-IR-8400S. ^{1}H-NMR spectra were obtained with Brukerspectrotometer model ultrashield at 300MHz in d6-DMSO solution with the TMS as internal standard.

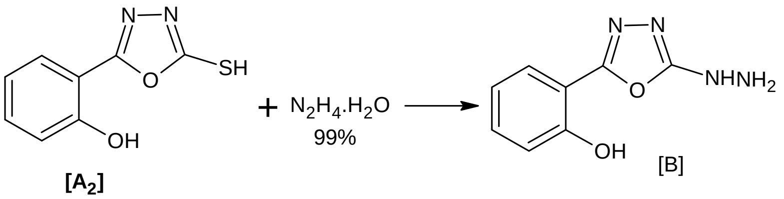
2.2. Preparation of [2-(5-mercapto-1,3,4-oxadiazole-2yl phenol] (A_2) compound:

The starting material [2-(5-mercapto-1,3,4-oxadiazole-2yl phenol] was prepared according to the method published in literature [7], scheme 1.



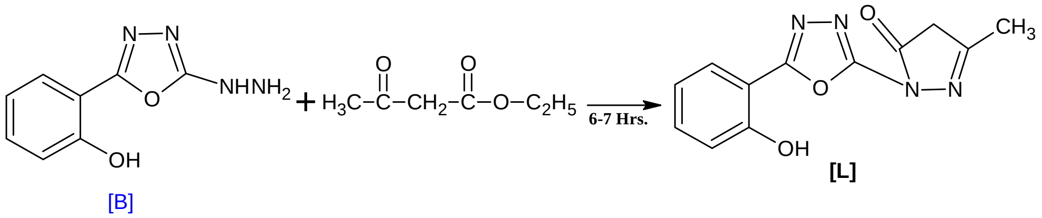
Scheme 1

0.388gm of [A_2] compound in (10ml) of ethanol absolute was added to (2mmol, 0.136g) of hydrated hydrazine (99% $N_2H_4.H_2O$), and then the mixture was refluxed for about 6hrs. on hot plate, the crude yellow precipitate of 2-[5hydrazino-1,3,4-oxadiazole]phenol [B] was collected after pouring the mixture poured in ice-water bath,filtered,washed with petroleum ether, dried in air and recrystallized from mixture of chloroform: ethanol(5:10ml.) to give yellow purified crystal, scheme 2.



2.3. Preparation of Ligand 2-[5-(2-hydroxy phenyl) 1,3,4-oxadiazol-2-yl]-5-methyl-2,4-dihydro-3*H*-pyrazol-3one [L]:

(0.01m, 1.92g) of [B] compound in 10ml of ethanol absolute was gradually added to (0.01m, 1.30g) of ethyl acetoe acetate in (5ml) of ethanol absolute and then the mixture was refluxed after stirring at room temperature for 15 minutes,then it was concentrated by evaporation on water bath until crude yellow precipitate was formed, filtered, washed several times with chloroformand acetone then dried in a desiccator overnight to afford 70% yield, scheme 3.



3. Results and Discussion

The physical properties of the prepared complexes were shwn in Table1.All the complexes were stable in air and not hygroscopic, soluble in most common organic solvents like ethanol,acetonitrile,DMF and DMSO.The results obtained from C.H.N elemental analyses were in agreement with the suggested formula of prepared complexes. The measurements of molar conductance for metal complexes solutions in DMF solvent displayed values of 70,149 and 166$\Omega^{-1}.cm^2mol^{-1}$ for Cr(III),Ni(II) and Cu(II) complexes respectively supporting the electrolytic behavior in 1:1 and 2:1 ratios[8],in contrast the Mn(II),Co(II) and Zn(II) complexes were neutral and recorded molar conductance fall in the range 15-30 $\Omega^{-1}.cm^2mol^{-1}$[10]

3.1.^{1}H-NMR Spectra:

The spectral data for the free ligand in DMSO-d^6 solution was reported along with the possible assignments in experimental. All the protons areat their expected region. The Ar-H, NH and CH=N proton signals, are shown in the regions of 6.36-7.9, 8.27 and 4.15 ppm, respectively,figure(1).

The weak signal at 11ppm could be assigned to the deshielded proton of phenolic -OH[11].The number of protons calculated from integration curves and the recorded chemical shifts in figure 1. Reveal the proposed structure of the formed new ligand [12].

3.2. G.C. Mass Spectra:

The gas chromatography-mass spectrum analysis is shown at figure(2), which provides vital information for elucidating the structure of the ligand its purity through the separation column enclosing data of retention relative to solvent (ethanol) used in preparation of standard solution.

The spectrum shows the molecular ion peak at m/e=258 and the isotopic peak in low abundance at m/e=259 (M^+) due to C^{13} and N^{15} isotopes. The base peak at m/e=120 is due to the fragment intensity of these peak reflects the stability and relative intersecting of the ion [13].

3.3. Infrared Spectra:

The disappearance of medium absorption at 3180cm^{-1} of [B] compound in the IR spectrum of free ligand confirms the ring closure in the presence of new bands of C=O,-OH,C=N-,N-N and –OH groups that were recognized in the structure of new ligand.

The IR spectrum of the ligand exhibits characteristic absorption at 1697cm^{-1}for $\upsilon_{C=O}$, as well as the strong absorption at 1166-1070cm^{-1} assigned to(N-N)mode of pyrazoline ring[14].As well as the broad band in the region 3400-3500 cm^{-1} may be attributed to hydrogen bonded –OH groups [14]. The spectra of all complexes show a broad absorptions in the regions(3600-3200)cm^{-1}, attributable to $\upsilon_{O\text{-}H}$ of the coordinated water molecules beside the rocking of O-H of coordinated water molecules[13-15].The imine–C=N- stretching frequency of the free ligand at1608cm^{-1} was undergone to downshift in all spectra of metal complexes in the regions(1500-1597)cm^{-1},this confirmed the participation of nitrogen atom of –C=N- of pyrazoline moiety in bonding with central metal ions[14-15].As well as,the negative shift in absorption of –C=O groupgivesa considerable investigation of coordination the metal ions through oxygen atom of carbony group in 2-position of pyrazoline ring that was formed in keto form isomer rather enol form in solid metal complexes[16].Furthermore,the far-infra-red spectra of all complexes exhibited weak to medium bands in the regions (440-506)cm^{-1} and (406-447)cm^{-1} to $\upsilon_{M\text{-}O}$ and υM-N bonds respectively [14].All the metal complexes except Ni(II)complex showed very weak absorptions in the region(280-366)cm^{-1}which may be attributed to M-Cl bonds,this support the coordination of chloride ions to metal ions[16-17].

3.5. Electronic Spectra and Magnetic Susceptibility Measurements:

The free ligand solution in ethanol spectrosol (10^{-3}M) concentration showed high intensity absorption in UV regions definitely at (32679, 37174 and 46082) cm^{-1} which may be assigned to ligand field and intra-ligand charge transfer transitions respectively, due to presence of chromospheres C=N, C=O of pyrazole and oxadiazole moieties [18].

The electronic spectral data of the free ligand and their metal complexes with Cr(III), Mn(II), Co(II), Ni(II), Cu(II) and Zn(II) ions complexes were recorded in ethanol as shown in Table 3.The chromium(III) complex solution in absolute ethanol showed three weak bands in regions(14124, 16129 and 18181) cm^{-1}, which are assigned to $^4A_2g(F)\rightarrow{}^4T_2g(F)$, $^4A_2g(F)\rightarrow{}^4T_1g$ and $^4A_2g\rightarrow{}^4T_1g(P)$ transitions, respectively that are constituent with an octahedral geometry around Cr(III) ion[17-18].The off white complex of manganese(II) complex in ethanol exhibited multiple weak bands at 18761 and 22222cm^{-1} that are assignable to $^6A_1g\rightarrow{}^4T_2g$ and $^6A_1g\rightarrow{}^4A_1g$,Eg (G) respectively, which associates with spin-forbidden transitions of high spin octahedral Mn(II) complex[19].

The cobalt(II) complex solution in ethanol displayed well resolved bands at 17550 and 20350 cm^{-1},that could be assigned to $^4T_1g(F)\rightarrow{}^4T_2g(F)$ and $^4T_1g(F)\rightarrow{}^4T_1g(P)$ transitions respectively confirming the geometry of high spin octahedral around Co(II) ion[20]The high intensity band at 29500cm^{-1} was assigned to the metal to ligand charge transfer[21],this supports the high delocalization between C=O,C=-N- groups with cobalt(II)ion and this could be refered to high covalency factor of Co-N and Co-O bonds in the formed complex[21]. The magnetic moment of 4.05B.M for the solid Co(II) complex.The nickel(II) complex displayed two bands in the visible region 25641 and 31347 cm^{-1}, which are assigned to$^1A_1g\rightarrow{}^1A_2g$ and $^1A_1g\rightarrow{}^1B_1g$ respectively and one band referred to overlapping of d-d transition with L→M C.T indicating the square planner environment around Ni(II) complex [20].

The electronic spectrum of copper(II) complex in ethanol showed low energy weak band at 16950 cm^{-1} and a high intensityone at 29630 cm^{-1}. The first band in visible region typically is expected for 10Dq that are corresponded to the $Eg^2\rightarrow{}^2T_2g$ transition[22-23] .

Moreover, the diamagnetic property (μ=0 B.M) of copper(II) complex may be considered as indicative of spin-spin coupling for binuclear Cu(II)-Cu(II) through the molecular association[23].

The magnetic moment values for Cr(III), Mn(II), Ni(II), Co(II), Cu(II) and Zn(II) complexes were shown in Table 4.

The complexes of Cr(III), Mn(II) and Co(II) complexes have magnetic moments fall in the range (3.45, 5.5 and 4.05)B.M respectively,this indicate that these complexes are typically high spin complexes and are constituents with expected values of d^3,d^5 and d^7 octahedral complexes[24].

3.6. Biological Activity:

The ligand in DMSO and its metal complexes were screened for their antibacterial and fungal activities at the same concentration and conditions similar to that of the tested complexes It is found obviously, Table 4 that metal complexes especially Zn(II) are potentially active against ***A. flavus*** and ***C. albican***. Also, it is found that all complexes except for (L), Mn(II) and Cr(II) didn't show antibacterial activity in comparison with tetracycline (32.5$\underline{+}$0.2), while all other complexes show high activity towards G^+ and G^- antifungal strains in range of (12.20$\underline{+}$0.5) for greater range compared with *Amphetamine* B (standard). The improved activity of metal complexes can be explained on the basis of chelation theory [25-26]. This theory explains that a decrease in the polarizability of the metal could enhance the lipophilicity of the complexes, which leads to abreakdown of the permeability of the cells, resulting in interference with normal cell processes [25-26].

This indicates that the chelation tends to make the ligand to act as more powerful and potent antimicrobial agents, thus, inhibiting the growth of bacteria and fungi more than the parent ligand (L).

The data in table 4 indicate that Zn(II) complex displayed the highest antimicrobial activity against G^+ and G^- bacteria and fungi under study, this was investigated on increasing of Lipophilic layer of these complexes and the chelation processes dominantly effects the biological behavior of the complexes that are potent against microbial and fungal strains.

4. Biological Activity:

The ***In-vitro*** biological activity of the investigated free ligand and its metal complexes were tested against the bacteria ***Escherichia coli*** and ***Staphylococcus aureus*** by disc diffusion method using nutrient agar as medium and streptomycin as control. The antifungal activities of the compounds were also tested by the well diffusion method against the fungi ***Aspergillusniger*** and ***Candida albicans***, on potato dextrose agar as the medum and *miconazole* as control. Each of the compounds was dissolved in DMSO and solutions of different concentrations (25, 50 and 100) ppm were prepared separately. In a typical procedure, a well was made on agar medium inoculated with microorganism. The well was filled with the test solution using a micropipette and the plate was incubated 24 hrs. for bacteria at 37°C and 72 hrs. for fungi at 30°C. during this period, the test solution diffused and the growth of the inoculated microorganism was affected the inhibition zone was developed, at which the concentration was noted. All determinations were performed in triplicate.

The bacteria sub. Cultures, ***Staphylococcus aureus*** and ***Escherichia coli*** were used for antibacterial test, whereas ***Aspergillusflavus*** and ***Candida albicans*** were used for antifungal test at the biology depentnat laboratories at Al-Mustansiriyah University, College of Science, using the modified Kirby-Baver disc diffusion method [8]. Briefly, 100 ml of the tested bacteria/ fungi were grown in 10 ml of freath media until they reached 105 cells ml^{-1} for bacteria and 102 cells ml^{-1} for fungi. 100 ml of microbial suspension was spread on the agar plate corresponding to the broth in which they were maintained.

The inhibition zone was measured by the disc method[9], the diameters of the inhibition zone were measured in millimeters. Standard disc of tetracycline (antibacterial agent) and Amphotericine is (antifungal agent) several as tre controls for antimicrobial activity. However, the filter discs impregnated with 10 ml of (DMSO solvent + D.W) were used as a negative (-ve) control. The used agar (Meuller-Hinton) was rigorously tested for composition and pH.

% R.I = 100 (A-B) / (C-B)

Where A= area if inhibition sample plate.
B= area of inhibition in the DMSO (control).
C= area of inhibition in the standard plate.

The ligand was compared to the standard with respect to negative (-ve),positive(+ve) grams of bacteria and fungi (two types of strands that were mentioned in Table2.

Conclusions

In the present work, a series of Cr(III), Mn(II), Co(II), Ni(II), Cu(II) and Zn(II) complexes with new ligand (L), have been prepared and characterized on the basis of elemental analysis,molar conductivity in DMF,magnetic susceptibility and FTIR and UV-Visible spectroscopy.All the complexes were octahedral geometries and are formed in 1:1 ratio except for Ni(II) complex which were in2:1(L:M),respectively. All complexes have been screened biologically against +ve and –ve Grams bacteria and fungi, it is found that most newly complexes showed remarkable activity against the tested microorganisms in comparison with standard *Tetracycline* and *Amphertoriaine* drugs.

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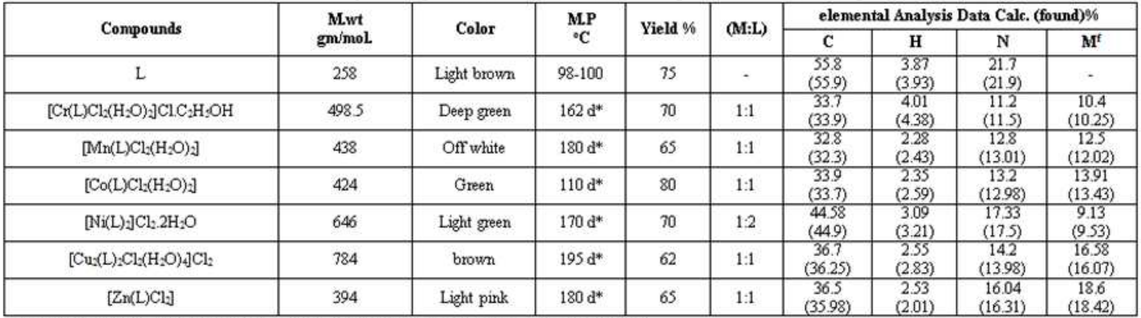
Table(1): The Physical Properties and Analytical Data of the Ligand (L) and its metal complexes



Compounds	M.wt gm/mol.	Color	M.P °C	Yield %	(M:L)	elemental Analysis Data Calc. (found)%			
						C	H	N	M^f
L	258	Light brown	98-100	75	-	55.8 (55.9)	3.87 (3.93)	21.7 (21.9)	-
$[Cr(L)Cl_2(H_2O)_2]Cl.C_2H_5OH$	498.5	Deep green	162 d*	70	1:1	33.7 (33.9)	4.01 (4.38)	11.2 (11.5)	10.4 (10.25)
$[Mn(L)Cl_2(H_2O)_2]$	438	Off white	180 d*	65	1:1	32.8 (32.3)	2.28 (2.43)	12.8 (13.01)	12.5 (12.02)
$[Co(L)Cl_2(H_2O)_2]$	424	Green	110 d*	80	1:1	33.9 (33.7)	2.35 (2.59)	13.2 (12.98)	13.91 (13.43)
$[Ni(L)_2]Cl_2.2H_2O$	646	Light green	170 d*	70	1:2	44.58 (44.9)	3.09 (3.21)	17.33 (17.5)	9.13 (9.53)
$[Cu_2(L)_2Cl_2(H_2O)_4]Cl_2$	784	brown	195 d*	62	1:1	36.7 (36.25)	2.55 (2.83)	14.2 (13.98)	16.58 (16.07)
$[Zn(L)Cl_2]$	394	Light pink	180 d*	65	1:1	36.5 (35.98)	2.53 (2.01)	16.04 (16.31)	18.6 (18.42)

d= decomposition point, and f=estimation of M% with flame atomic absorption spectroscopy

Table (2): Antibacterial Activity of Ligand and its Metal Complexes

Compound	**Inhibition *Escherichia coli***		***Staphy. aurea***	***Fungi***
	G^+	G^-	Asp.	Can.
L	15.9±0.3	20.5±0.1	18.3±0.1	16.0±0.1
$[Cr(L)Cl_2(H_2O)_2]Cl\,.EtOH$	18.32±0.1	21.0±0.1	14.3±0.1	15.11±0.1
$[Co(L)Cl_2(H_2O)_2]$	33.51±0.2	23.51±0.1	10.91±0.2	13.11±0.1
$[Cu_{2(}L)_2Cl_2(H_2O)_4]Cl_2$	35.22±0.11	21.11±0.2	17.51±0.3	10.9±0.1
$[Mn(L)Cl_2(H_2O)_2]$	25.41±0.31	18.95±0.1	17.05±0.3	12.0±0.1
$[Ni(L)_2]Cl_2$	31.45±0.11	17.51±0.2	14.71±0.2	15.0±0.1
$[Zn(L)Cl_2]$	35.91±0.25	22.17±0.1	20.11±0.1	18.99±0.1

Control DMSO= O

Antibacterial reagent	**Antibacterial**		***Antifungal***	
	G^+	G^-	G^+	G^-
Tetracycline	32.51±0.2	30.11±0.25	-	-
Amphoteriaine	-	-	19.89±0.2	20.5±0.1

G^+, G^- = Gram positive and negative of bacteria and Fungi.

Table (3): The Characteristic Stretching Vibration Frequencies (cm^{-1}) Located at FT-IR of (L) and its Metal Complexes

Compounds	$\nu_{C=O}$	$\nu_{C=N}$	ν_{N-N}	ν_{M-N}	ν_{M-O}	ν_{M-Cl}
L	1697 (s) 3220	1608(s)	1070 (m)	-	-	-
$[Cr(L)Cl_2(H_2O)_2]lCl.C_2H_5OH$	1653 (s) 3440	1597(s)	1082 (m)	499 (w)	416 (m)	350 (vw) 833^a
$[Mn(L)Cl_2(H_2O)_2]$	1060 (s) 3560	1500(s)	1072	492 (w)	420 (m)	366 (vw) 850a
$[Co(L)Cl_2(H_2O)_2]$	1649 (s) 3600	1514(s)	1078 (m)	476(m)	435(w)	320 (vw) 844^a
$[Ni(C_{12}H_{10}N_4O_3)_2]Cl_2.2H_2O$	1610 (s) 3266	1598(s)	1080 (m)	494(m)	457(w)	-
$[Cu_2(C_{12}H_{10}N_4O_3)_2Cl_2(H_2O)_4]Cl_2$	1608 (s) 3489	1543 (s)	1074	506(m)	447(w)	280 (w) 835^a
$[Zn(C_{12}H_{10}N_4O_3)Cl_2]F$	1650 3590(br.)	1535	1083	440(m)	445(w)	330

Where : s=strong, m=medium, w=weak and vw= very weak and a=rocking of O-H for coordinated water molecules.

Table(4): Electronic Spectra, molar conductance in (DMF) and magnetic moments (B.M) for (L) and its metal complexes

Compound	**Absorption cm^{-1}**	**UV-Vis.(cm^{-1})**	**Molar Cond. $\Omega^{-1}.cm^2mol^{-1}$**	**μeff. B.M**	**Geometry**
L	46082 37174 32679	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$ C.T	-	-	-
$[Cr(L)Cl_2(H_2O)_2]Cl.EtOH$	14124 16129 18181	${}^4A_2g \xrightarrow{\nu 1} {}^4T_2g$ ${}^4A_2g \xrightarrow{\nu 2} {}^4T_1g$ ${}^4A_2g \xrightarrow{\nu 3} {}^4T_1g$ (P)	70	3.45	Oh
$[Mn(L)Cl_2(H_2O)_2]$	18761 22222	${}^6A_1g \xrightarrow{\nu 1} {}^4T_2g$ ${}^6A_1g \xrightarrow{\nu 2} {}^4A_1g, {}^4Eg(G)$	20	5.5	Oh
$[Co(L)Cl_2(H_2O)]$	17550 20350 29500	${}^4T_1g \xrightarrow{\nu 1} {}^4T_2g$ ${}^4T_1g \xrightarrow{\nu 2} {}^4A_2g$ M→L C.T	15	4.05	Oh
$[Ni(L)_2]Cl_2.2H_2O$	25641 31347	${}^1A_1g \xrightarrow{\nu 1} {}^1B_1g$ ${}^1A_1g \xrightarrow{\nu 2} {}^1B_2g$	166	0	S.P
$[Cu_2(L)_2Cl_2(H_2O)_4]Cl_2$	16950 29630	$Eg^2 \rightarrow Tg^2$ ${}^2B_1g \xrightarrow{\nu 2} {}^2A_2g$ L→M C.T	149	0	Oh
$[Zn(L)Cl_2]$	30550	L→M C.T	30	0	Th

$L=C_{12}H_{10}N_4O_3$ formula of the free ligand,Oh.=octahedral ,Th.=tetrahedral and s.p=square planner geometries.

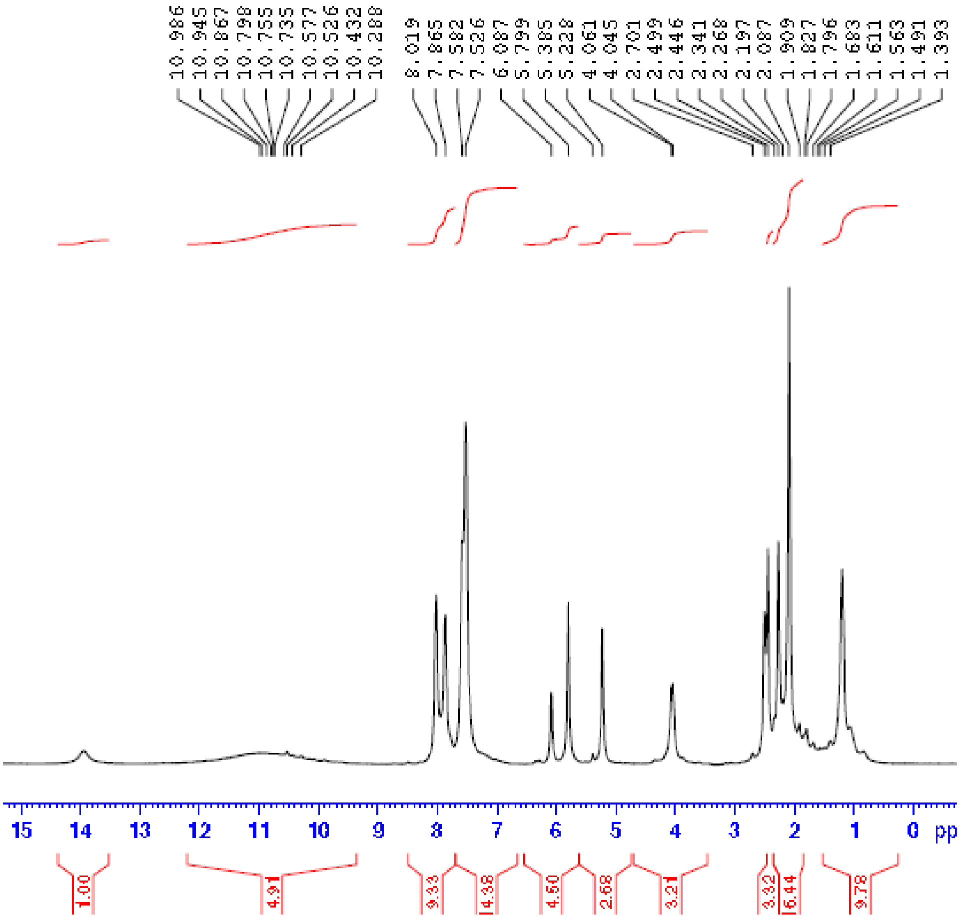


Figure 1: H^{1}NMR of Ligand (L) in d^6-DMSO Solution

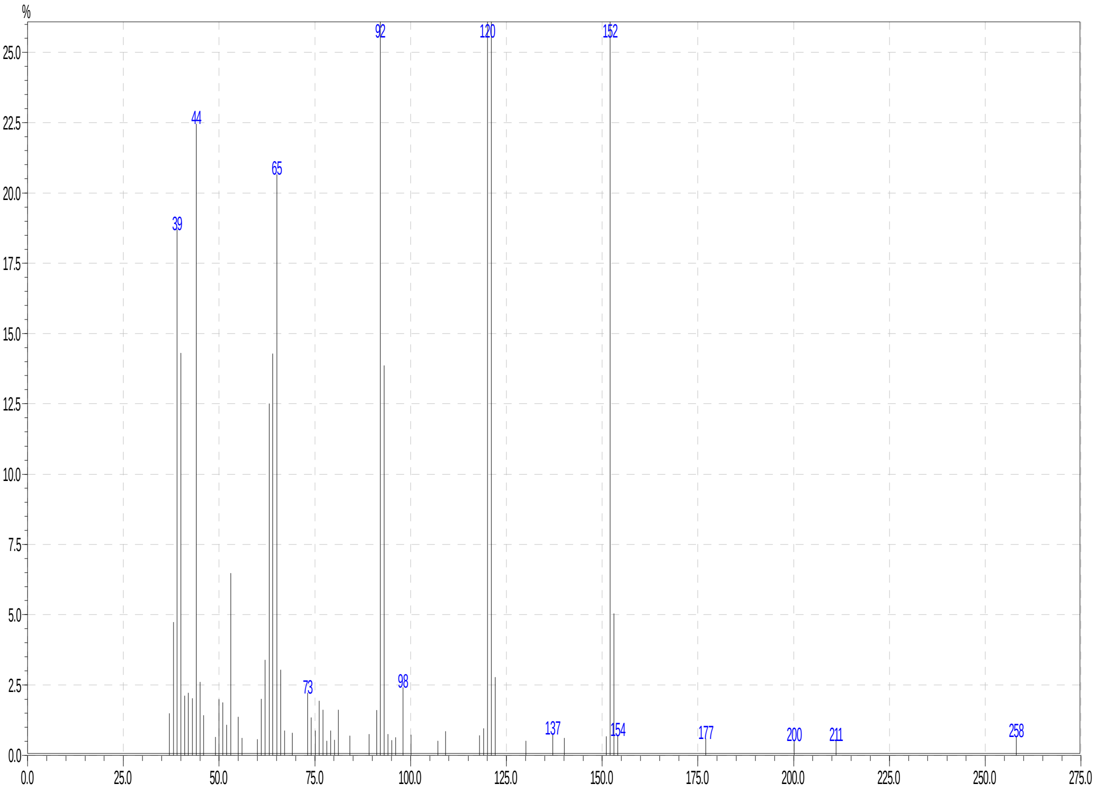


Figure 2: GC-Mass Spectrum of Ligand (L)

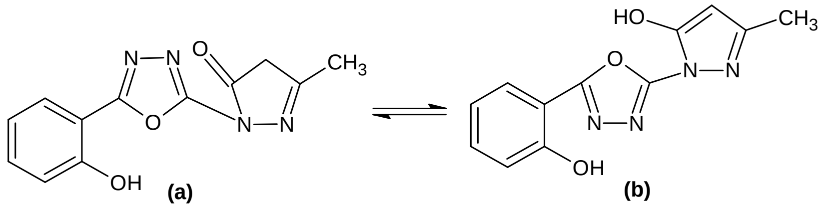
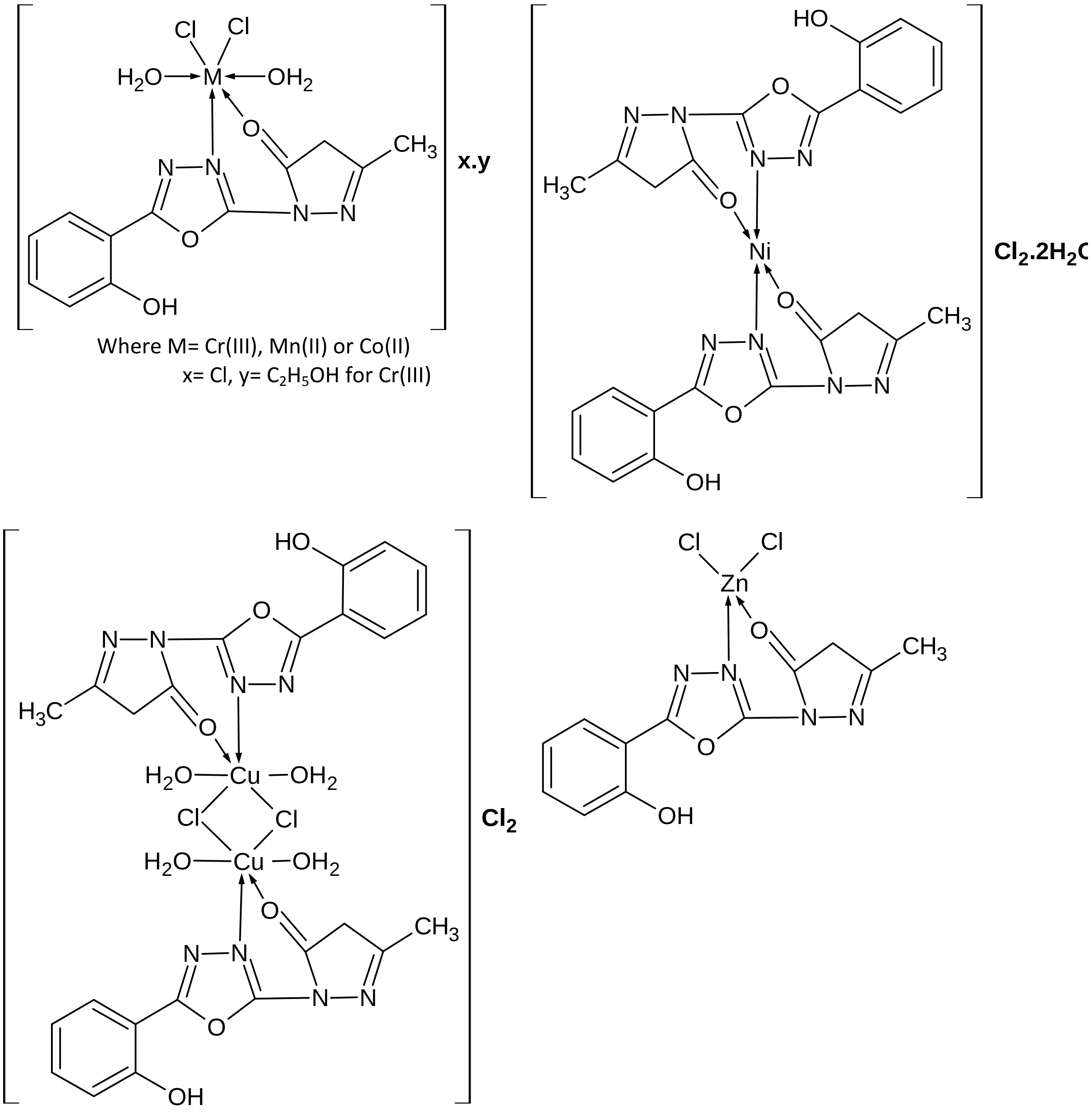


Figure (3):Tautomerism structures of ligand



Where M= Cr(III), Mn(II) or Co(II)
x= Cl, y= C_2H_5OH for Cr(III)

Figure(4)-stereochemical structures of metal complexes

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