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Synthesis and characterization of some new metals complexes of ((2-Chloro acetyl) carbamothioyl) glycine

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Abstract---A new ligand ((2-Choroacetyl) carbamothioyl) Glycine (CCG) is synthesized by reaction of (Chloro acetyl isothiocyanate) with Glycine (1:1). It is characterized by micro elemental analysis (C.H.N.S.), FT-IR, (UV-Vis) and 1H and 13CNMR spectra. Some metals ions complexes of this ligand were prepared and characterized byFT-IR,UV-Visible spectra, conductivitymeasurements, magnetic susceptibility and atomic absorption. From results obtained, the following formula [M(CCG)2] where M2+ = Mn, Co, Ni, Cu, Zn, Cd and Hg, the proposed molecular structure for these complexes as tetrahedral geometry, except copper complex is has square planer geometry.

Keywords--- glycine, chloro acetyl isothiocyanate, complexe.

Introduction

Glycine acts as a bidentate ligand in its coordination with metal ions and stabilizes the complexes via hydrophobic interaction. The glycine to type of association serves as a basis of understandingthe nature of metal aminoacids interaction. The essential metals such as iron,manganese, cobalt and nickel play vital various roles in biological processes in form of complexes and theirstability contributes to their efficiency. The rolesplayed by these metals include catalysing enzymaticprocess, storage, checking of toxicity of metals at certain concentration [1-3]. Three novel mixed ligand metal complexes have been synthesized by the reaction ofZn(II), Sn(II), and Ce(III) with gemifloxacin (GMFX) in the presence of glycine (Gly) (1:1:1 molarratio). The coordination possibility of the two ligands toward metal ions has been proposed in the light of elemental analysis, molar conductance, spectral infrared (IR),

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ultraviolet-visible (UV-Vis) and proton-nuclear magnetic resonance (^{1}HNMR), and magnetic studies. Results suggest that GMFX and Gly interact with the metal ions as bidentate ligands. Electronic and magnetic data proposed the octahedral structure for all complexes[4]and als Hassan Mansouri-Torshizia and co- work synthesisTwo novel, neutral and water soluble Pd (II) complexes of formula [Pd(Gly)(Ala)] [1]and [Pd(Gly)(Val)] [2] (Gly, Ala, and Val are anionic forms of glycine, alanine, and valine amino acids, respectively) and characterized by FT-IR, UV–Vis, ^{1}HNMR, elemental analysis, and molar conductivity measurement.The data revealed that each amino acid binds to Pd(II) through the nitrogen of –NH2 and the oxygen of –COO– groups and acts as a bidentate chelate. The aim of this work is preparation some new metal ions complexes of [((2-Chloro acetyl) carbamothioyl) Glycine (CCG)

Materials and Methods

(Chloroacetyl chloride), (Glycine) (Fluka), Manganese chloridetetra hydrate ($MnCl_2.4H_2O$), Cobalt chloride hexahydrate ($CoCl_2.6H_2O$), Nickelchloride hexahydrate ($NiCl_2.6H_2O$), Copper chloride dehydrate ($CuCl_2.2H_2O$), Zinc chloride ($ZnCl_2$), Cadmium chloride hydrate ($CdCl_2.H_2O$) and Mercury chloride ($HgCl_2$). All reagents were analar or chemical pure grade by BDH, Merck and Fluka.

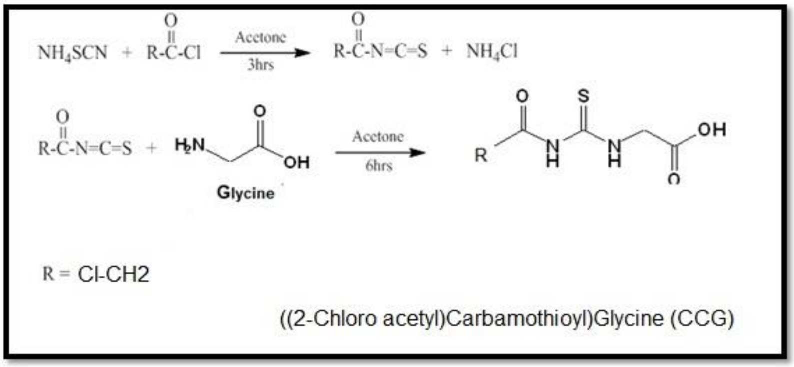
Instruments

HNMR,^{13}CNMR was recorded using Ultra Shield 300 MH$_z$ Switzerland at sharif university of techonlg. Melting point was recorded by using Stuart- melting point apparatus. FT-IR spectra were recorded as KBr disc using 3800 Shimadzu in the range of 4000-400 cm^{-1}. Electronic spectra were obtained using UV-160 Shimadzu spectra photometer at 25 °C in (1x10^{-3}) M DMSO. Conductivity was measured by using Philips Pw. Digital. Micro elemental analyses (C.H.N.S) were performed using acrlo Erba1106 elemental analyzer. Magnetic susceptibility measurements were obtained by balance magnetic susceptibility by model MSB-MKI. Metal contents of the complexes were determined by atomic absorption technique by using Shimadzu (AA680G).

Preparation of Ligand (CCG)

Preparation of the (Chloro acetyl isothiocyanate**)** [5] Mixture of Chloro acetyl chloride (2.1 ml, 1mmol) and ammonium thiocyanate (2g, 1mmol) in (25ml) of acetone was stirred under refluxed for 3 hours and then filtered, the filtrate was used for further reaction

Preparation of (2-Chloro acetyl) carbamothioyl) Glycine **(CCG)** (2g, 1mmol) of Glycine in (20ml) acetone was rapidly added to the maintain vigorous reflux. After refluxing for 6 hours, the resulting solid was collected, washed with acetone and recrystallization from ethanol. Scheme (1), Yield (83%), (m.p =118-120) °C, C% found (28.50) while calculate (28.80), H% found (3.30) while calculate (3.88), N% found (13.30) while calculate e (13.45), S% found (15.20) while calculate (15.50).(Scheme(1)



Scheme 1. Preparation of Ligand (CCG)

Synthesis of metal complexes

(0.4210 mmole) of (CCG) was dissolved in 25ml of ethanol containing (0.112g, 2mmole) of KOH, then the solution of following metal salts $MnCl_2.4H_2O$ (0.2g, 1mmole), $CoCl_2.6H_2O$ (0.24g, 1mmole), $NiCl_2.6H_2O$ (0.24g,1mmole), $CuCl_2.2H_2O$ (0.2g, 1mmole), $ZnCl_2$(0.14g,1mmole), $CdCl_2.H_2O$ (0.2g,1mmole), and $HgCl_2$ (0.3g,1mmole) in ethanol, were added dropwise to the solution of(CCG^-K^+).The precipitate formed immediately after stirring the mixture at room temperature for 3hours. The precipitate was collected by filtration, washed with distilled water and ethanol and dried under vacuum. Physical properties were given in Table (1).

Results and Discussion

The solid complexes were soluble in some common solvent such as dimethyl formamide,dimethyl sulphoxide, and relatively thermally stable. The molar conductivity of all complexes in DMSO were found to be non-electrolyte, Table (1) includes the physical properties for Ligand(CCG) and its metal complexes.

Table 1
Physical properties of (CCG) and its metal complexes

Compund	M.wt (gm/mol)	Color	M.p °C or dec.	M% Calculation (Found)	Molar condu. $Ohm^{-1}Cm^2mol^{-1}$	μ_{eff} (B.M)
(CCG)	210.5	Orange	118-120	-	2.9	-
[Mn$(CCG)_2$]	474	Deep Brown	220- 222	11.60 (11.51)	3.96	5.86
[Co$(CCG)_2$]	478	Deep Green	253 Dec.	12.34 (12.22)	13.6	4.44
[Ni$(CCG)_2$]	477	Brown	225 Dec.	12.16 (12.06)	3.6	3.09
[Cu$(CCG)_2$]	482	Green	213-215	13.07 (12.98)	4.0	1.74

[Zn(CCG)$_2$]	484	Deep Brown	200-202	13.43 (13.15)	14	0
[Cd(CCG)$_2$]	531.4	Deep Brown	50-52	21.15 (21.02)	3.45	0
[Hg(CCG)$_2$]	620	Brown	251-253	32.42 (32.35)	2.64	0

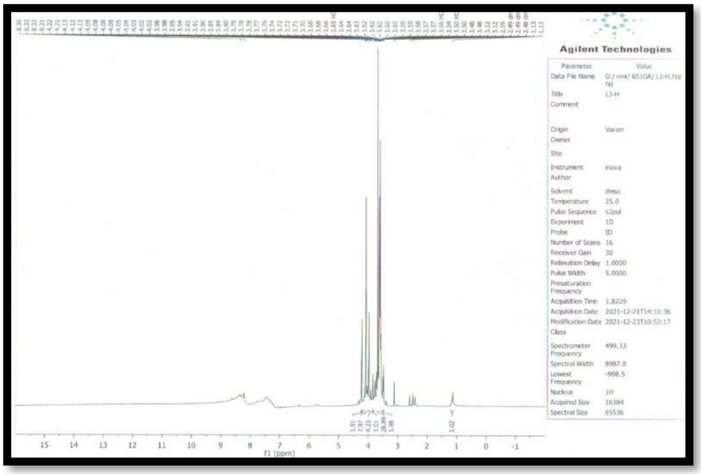
Spectral studies

^{1}H and ^{13}C-NMR spectra

^{1}H-NMR spectrum for (CCG) in DMSO as solvent, Fig. (1) showed the following signals:singlet atδ (4.21)ppm for (2H,Cl-CH$_2$), singlet at δ(4.22) ppm for (2H, CH$_2$), multiplet atδ(2.48-2.49) ppm for DMSO, singlet at δ(8.21) ppm for (1H,NH sec.amine), singlet at δ(8.36) ppm for (1H, NH sec.amide), singlet at δ(8.32) ppm for (1H, COOH) [6] Table (2)

Table 2
^{1}HNMR) Spectral data for ligand (CCG)

Compound	No of protens	(ppm)δ
Ligand (CCG)	S (2H,ClCH$_2$)	4.21
	S (2H,CH$_2$)	4.22
	2d (1H,NH$_{Sec,}$amine)	8.21
	s (1H,COOH)	8.32
	S (1H,NHsec,amide)	8.36



Parameter	Value
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Comment	
Origin	Varian
Owner	
Site	
Instrument	inova
Author	
Solvent	dmso
Temperature	25.0
Pulse Sequence	s2pul
Experiment	1D
Probe	ID
Number of Scans	16
Receiver Gain	30
Relaxation Delay	1.0000
Pulse Width	5.0000
Presaturation Frequency	
Acquisition Time	1.8229
Acquisition Date	2021-12-21T14:10:36
Modification Date	2021-12-23T10:52:17
Class	
Spectrometer Frequency	499.33
Spectral Width	8987.8
Lowest Frequency	-998.5
Nucleus	1H
Acquired Size	16384
Spectral Size	65536

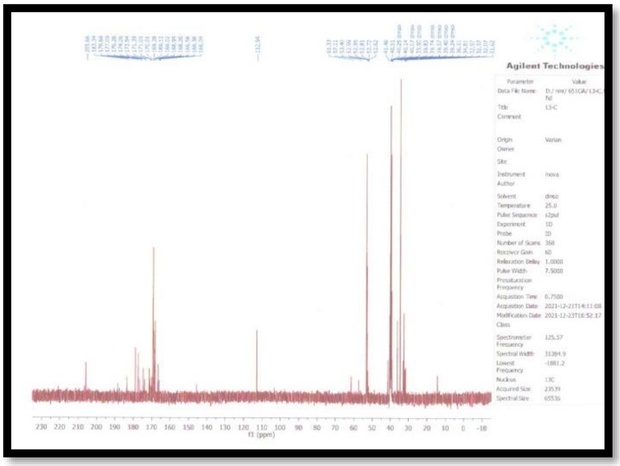
Fig. 1. ^{1}HNMR spectrum of (CCG)

2) The ^{13}C-NMR spectrum for Ligand (CCG) in DMSO, Fig. (2) showed the following signals:signals at δ (41.46)ppm for Cl-CH$_2$, signals at δ(52.62) ppm for (CH$_2$), multiplet at δ(39.24-40.25) ppm for DMSO,signal at δ(171.39) ppm for

(C=O sec.amide), signal at δ(173.54) ppm for (COOH), signal at δ(183) ppm for (C=S).

Table 3
^{13}CNMR Spectral data for ligand (CCG)

Compound	No of protens	δ(ppm)
Ligand (CCG)	s(Cl-CH_2)	41.46
	$_s$(CH_2)	52.62
	s(C=$O_{sec.amide}$)	171.39
	(C OOH) s	173.54
	s (C=S)	183



Parameter	Value
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Comment	
Origin	Varian
Owner	
Site	
Instrument	inova
Author	
Solvent	dmso
Temperature	25.0
Pulse Sequence	s2pul
Experiment	1D
Probe	ID
Number of Scans	368
Receiver Gain	60
Relaxation Delay	1.0000
Pulse Width	7.5000
Presaturation Frequency	
Acquisition Time	0.7500
Acquisition Date	2021-12-21T14:11:08
Modification Date	2021-12-23T10:52:17
Class	
Spectrometer Frequency	125.57
Spectral Width	31384.9
Lowest Frequency	-1881.2
Nucleus	13C
Acquired Size	23539
Spectral Size	65536

Fig. 2. ^{13}CNMR spectrum of Ligand (CCG)

Infrared spectra

FT-IR spectrum of the free Ligand (CCG), Fig. (3), showed bands due to amido υ(NH), υ(C=O) and υ(C=S) which absorbed at (3414) cm^{-1}, (1620) cm^{-1}, and (1230) cm^{-1} respectively, while another absorption band appeared at (1724) cm^{-1} could be explained as υ$(COO)_{asym}$ [7] , [8] where the υ$(OCO)_{sym}$ was noticed at (1404) cm^{-1}. The FT-IRspectra of complexes exhibited marked difference between bands belonging to the stretching vibration of υ(NH) of the amine group in the range between (3390 -3336) cm^{-1} shifted lower frequencies by (24-78) cm^{-1} suggesting of the possibility of the coordination of (CCG) through the nitrogen atom at the amine group[9]. Absorption assigned for υ$(COO)_{asym}$ was noticed at the range (1651-1570) cm^{-1} shifted to higher fre frequencies by (73-154) cm^{-1} while the band caused by υ$(COO)_{sym}$ appeared between (1485-1438) cm^{-1} shifted to lower frequencies by (81-34) cm^{-1} which indicates to the coordination of the carboxylic group to the central ion[10] The stretching vibration band υ(C=O) and υ(C=S) carbonyl group either shows no change or very little in their frequencies (1624-1616) cm^{-1} and (1238-1230) cm^{-1} respectively there for indicating do not coordinate to the metal ion [11] . Metal- nitrogen and metal-oxygen bonds were confirmed by the presence of the stretching vibration of υ(M-O) and υ(M-N) around (509-474) cm^{-1} and (412-462) cm^{-1} respectively. Table (4) describes the important bands and assignment for free (CCG) and its complexes.The FTIR spectrum of(Ni)complex showed in Fig. (3).

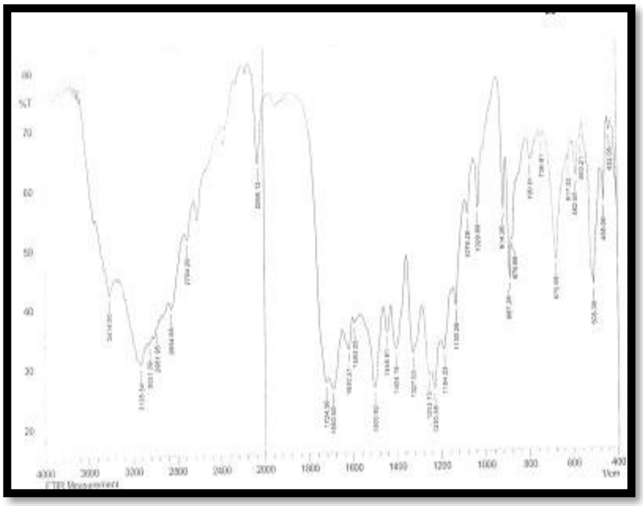

Fig. 3. FT-IR spectrum of Ligand (CCG)

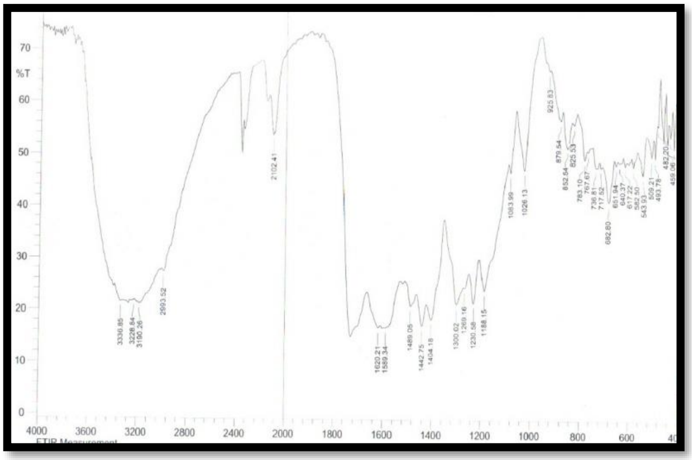

Fig. 4. FT-IR spectrum of Complex [Ni(CCG)$_2$]

Table 4
Some FT-IR frequencies in (cm^{-1}) forLigand (CCG) and its metal complexes

Complex	υ(N-H)	υ(COO)$_{sym}$	υ(COO)$_{asym}$	υ(C=O)	υ (C=S)	υ(M-O)	υ (M─N)
(CCG)	3414(m) 3128(m)	1404(m)	1724(m)	1620(m)	1230(s)	-	-
[Mn(CCG)$_2$]	3367(m)	1438(m)	1585(m)	1618 (m)	1233(w)	509(w)	462(w)
[Co(CCG)$_2$]	3390(s)	1458(w)	1639(s)	1616(s)	1234(w)	474(w)	432(w)
[Ni(CCG$_2$]	3336(m) –	1442(m)	1589(m)	1620(m)	1230(m)	482w)	459(w)

[Cu(CCG)$_2$]	3390(m)	1485(m)	1651(w)	1618(m)	1234(m)	474(w)	459(w)
[Zn(CCG)$_2$]	3352(m) _	1438(s)	1570(w)	1624(m)	1235(w)	486(w)	424(w)
[Cd(CCG)$_2$]	3336(m)	1485(s)	1651 (m)	1618(m)	1238(s)	493(w)	459(w)
[Hg(CCG)$_2$]	3388(m) _	1438(m)	1635(m)	1616(m)	1230(w)	497(w)	412(w)

b = browed, w = weak, s = strong, m = medium

Magnetic properties for the metal complexes

Magnetic moment (ì$_{eff}$) with regard to complexes related to Mn^{+2}(d^5), also Co^{+2}(d^7) have been indicated (5.86)B.M, also (4.44)B.M, that in anticipated spin-only values Higher value related to ìeff of Ni^{+2}(d^8) complex (3.09) B.M because of orbital contributions[12] Magnetic moment ìeff related to Cu^{+2}(d^9)complex indicated (1.74)B.M in expected value to one electro[13] , all data are indicated in the Table(1)

Electronic spectral

The UV-Visible spectra of Ligand(CCG) and its complexes recorded in Table (5), the solution of (CCG) in 10^{-3}M (DMSO) exhibited two peaks Fig. (5) at (38022) cm^{-1} and (33444) cm^{-1} which are attributed to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transition respectively[14]

The spectra of complexes

[Mn(CCG)$_2$] d^5: The brown complex of Mn (II) shows bands at (37878) cm^{-1}and (28169) due to ligand felid and the electronic transfer$^6A_1 \rightarrow {}^4T_2$ and another band at (19084) cm^{-1} which is caused by the electronic transfer$^6A_1 \rightarrow {}^4T_{1\,G}$ [15]

[Co(CCG)$_2$] d^7: The spectrum of the deep green complex gave four bands at, (34013) cm^{-1},(28901)cm^{-1}, (22727) cm^{-1} and (16920) cm^{-1} and (10526) cm^{-1} attributed to ligand felid , (C.T), $^4A_2 \rightarrow {}^4T_{1(P)}$, $^4A_2 \rightarrow {}^4T_{1(F)}$ and $^4A_2 \rightarrow {}^4T_{2(F)}$ respectively, and the rach interelectronic repulsion parameter (B$^-$) was found to be (537.9) cm^{-1}, from the relation $\beta = B^- / B_0$, was found to be a equal (0.522), these parameter are accepted to Co(II) tetrahedral complex[16]

[Ni(CCG)$_2$] d^8: The spectrum of Brown complex of Ni (II) has revealed the following electronic transfer ligand felid , (C.T),mix with $^3T_{1(F)} \rightarrow {}^3T_{1(P)}$, $^3T_{1(F)} \rightarrow {}^3A_{2(F)}$ and $^3T_1 \rightarrow {}^3T_{2(F)}$, transition at (33444)cm^{-1},(29069) cm^{-1}, (24390) cm^{-1} and (18018) cm^{-1} and (11013) cm^{-1} respectively, the (B$^-$) value found to be (624.6) cm^{-1}, while β was equal to (0.60) these are the characteristics for tetrahedral complexes of Ni (II) [17]

[Cu(CCG)$_2$] d^9: The spectrum ofgreen complex of Cu(II) Fig. (6) show three bands at (37037) cm^{-1}, (33222) cm^{-1} and (28901) cm^{-1} and (11037) cm^{-1} caused to ligand felid ,(C.T), $^2B_{1g}\rightarrow{}^2A_{1g}$ and $^2B_{1g}\rightarrow{}^2B_{2g}$ transition respectively[18]

The complexes of **[Zn(CCG)$_2$]**, **[Cd(CCG)$_2$]** and **[Hg(CCG)$_2$]** shows only ligand felid and charge transfer of (M→L) at (33557) cm^{-1},(34364,28985) cm^{-1} and (36496,28901) cm^{-1} respectively [19] , All transition with their assignments are summarized in Table (5).

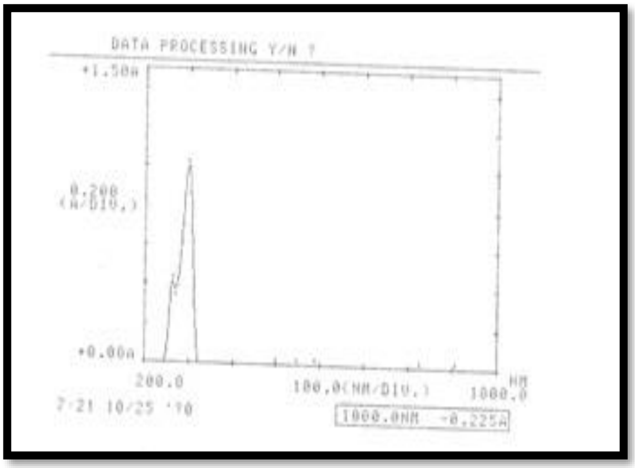

Fig. 5. Electronic spectrum of ligand(CCG)

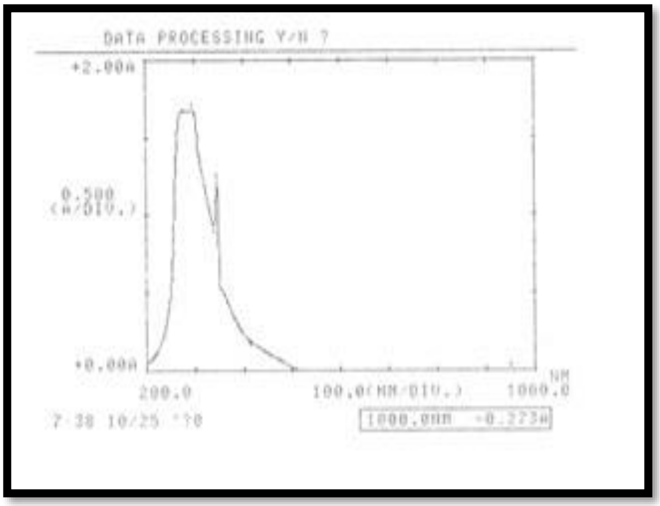

Fig. 6. Electronic spectrum of [Co(CCG)$_2$]

Table 5
The peaks electronic transitions and structure geometries of Ligand(CCG)and its complexes

الملاحظات	$_{max}\epsilon$ L.mol^{-1} .cm^{-1}	Wave number cm^{-1}	ABC	λ_{max} (nm)	Complex	No
$\pi\rightarrow\pi^*$	410	38022	0.410	263	)الليكاند(CCG	1
$n\rightarrow\pi^*$	1000	33444	1.000	299		
L.F	549	37878	0.549	264	]Mn(CCG)$_2$[	2
$^6A_1\rightarrow{}^4T_2(G)$	180	28169	0.180	355		
$^6A_1\rightarrow{}^4T_1(G)$	35	19084	0.035	524		
L.F	1676	34013	1.676	294	]Co(CCG)$_2$[	3
C.T	1220	28901	1.220	346		
$^4A_2\rightarrow{}^4T_{1(p)}$	115	22727	0.115	440		
$^4A_2\rightarrow{}^4T_{1(f)}$	38	16920	0.038	591		
$^4A_2\rightarrow{}^4T_2$	25	10526	0.025	950		
L.F	2196	33444	2.196	299	]Ni(CCG)$_2$[	4
C.T	1922	29069	1.922	344		
$^3T_{1(f)}\rightarrow{}^3T_{1(p)}$	188	24390	0.188	410		
$^3T_{1(f)}\rightarrow{}^3A_2$	30	18018	0.030	555		
$^3T_{1(f)}\rightarrow{}^3T_{2\ (f)}$	25	11013	0.025	908		
L.F	1293	37037	1.293	270	]Cu(CCG)$_2$[	5
C.T	1188	33222	1.188	301		
$^2B_{1(g)}\rightarrow{}^2A_{1(g)}$	1139	28901	1.139	346		
$^2B_{1(g)}\rightarrow{}^2B_{2(g)}$	30	11037		906		
L.F	1481	33557	1.481	298	]Zn(CCG)$_2$[	6
L.F	2047	34364	2.047	291	]Cd(CCG)$_2$[	7
C.T	1374	28985	1.374	345		
L.F	1485	36496	1.485	274	]Hg(CCG)$_2$[	8
C.T	1241	28901	1.241	346		

L.F=Ligand felid
C.T = Charge transfer

According to spectral data as well as those obtained from elemental analyses, the chemical structure of the complexes may be suggested as tetrahedral for [M(CCG)$_2$] where M^{2+} = (Mn, Co, Ni, Zn, Cd and Hg), Fig. (7) while Copper complex s has square planer

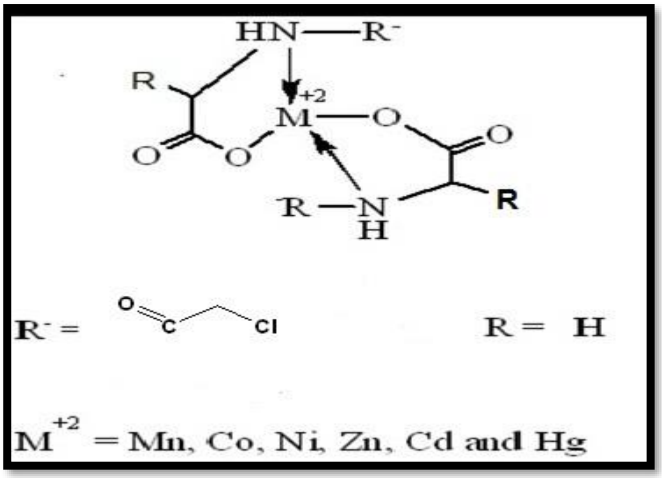

Fig. 7. Suggested structure of complexes [M(CCG)$_2$]

Conclusion

The new ligand in the presented study was prepared through reaction from the 2-Chloro acetyl isothiocyanate with Glycine, ligand has been characterized through elemental micro analysis C.H.N.S., FTIR, UV Vis also ^{1}H,^{13}C-NMR spectra. Ligand's metal complexes have been prepared, also characterized through FTIR, UV Vis spectra, magnetic measurements, conductivity measurements, the suggested geometrical structure with regard to complexes have been tetrahedral geometry except for the copper complex that has square planer

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