

Synthesis and Characterization of Some New Metals Complexes of ((4-Methoxy Benzoyl) Carbamothioyl) Glycine

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ABSTRACT

A new ligand (4- methoxy benzoyl) carbamothioyl) Glycine (MCG) is synthesized by reaction of (4- methoxy benzoyl isothiocyanate) with Glycine(1:1). It is characterized by micro elemental analysis (C.H.N.S.), FT-IR, (UV-Vis) and ¹H and ¹³C-NMR spectra. Some metals ions complexes of this ligand were prepared and characterized by FT-IR, UV-Visible spectra, conductivity measurements, magnetic susceptibility and atomic absorption. From results obtained, the following formula [M(MCG)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 4 methoxy benzoyl isothiocyanate, complexes

INTRODUCTION

Glycine (Gly; NH₂CH₂COOH) is the simplest amino acid, consisting of a single carbon bonded to carboxyl and amino groups, and is the only amino acid that does not have optical isomers. In the human body, Gly contributes to building muscle protein [1], absorbing calcium [2-4], producing antibodies [5 -7], enzymes [8-10], and hormones [11, 12], and recovering from physical injury [13-14], complexation between amino acids and transition metal ions has received much attention due to their broad and increasing number of applications, including as antibacterial agents with potent biological activity (15-19). Therefore, it has become one of our most important trends, is to obtain different derivatives of amino acids with transitional elements,

novel series of transitional metal complexes of Cu(II), Co(II), Ni(II), Zn(II) and Fe(II) with N-modified glycine as ligand provide from phenylserinol were synthesized and characterized via elemental analysis, MS, IR, UV-VIS thermogravimetric analysis and ESR. The thermal stability of the ligand and synthesized complexes was discussed in the 20-1000oC temperature range. The results indicate that the organic compound acts as a bidentate ligand, its coordination involving the carboxylate oxygen and the nitrogen atom [20] and also The binary and ternary complex systems of Fe(III), Pb(II), Co(II), Al(III), La(III), Sr(II), Cr(III), Ti(II), Zr(IV) and Th(IV) with sulfathiazole (as the primary ligand) and the amino acid glycine (as the secondary ligand) have been assessed potentiometrically at a temperature of 25.0±0.1°C and a concentration of 0.1 M NaClO₄ in a 25% (v/v) ethanol–water solution. To investigate the effect of the secondary ligand on the formation of 1:1 M:Sulfathiazole, the stoichiometries and stability constants of binary complexes consisting of the above metal ions in a 1:1, 1:2 and/or 1:3 ratio were assessed. The protonation constants of the complexes were measured for the M:Sulfathiazole:Glycine system at a 1:1:1 ratio. In the case of Al(III) and Th(IV), glycine as a secondary ligand prefers to bind with a [M–Sulfathiazole] binary complex rather than to the metal ion complex in an aqueous solution. In all cases, the stability order of the binary (M:Sulfathiazole) and ternary (M:Sulfathiazole:Glycine) complexes was examined.[21], Asma S. Al-Wasidi and co- worker [22] report findings from the synthesis and IR-based characterization of Zn(II)-, Cu(II)-, Ni(II)-,

Fe(III)-, Mn(II)-, and Cr(III)-Gly complexes and the preparation of ZnO, CuO, NiO, Fe₂O₃, MnO₂, and Cr₂O₃ using solid-state thermal

decomposition of these complexes. The aim of this work is preparation some new metal ions complexes of [4- methoxy benzoyl) carbamothioyl) Glycine (MCG)

MATERIALS AND METHODS

(4-methoxy benzoyl chloride), (Glycine) (Fluka), Manganese chloride tetrahydrate (MnCl₂.4H₂O), Cobalt chloride hexahydrate (CoCl₂.6H₂O), Nickel chloride hexahydrate (NiCl₂.6H₂O), Copper

chloride dehydrate (CuCl₂.2H₂O), Zinc chloride (ZnCl₂), Cadmium chloride hydrate (CdCl₂.H₂O) and Mercury chloride (HgCl₂). All reagents were analar or chemical pure grade by BDH, Merck and Fluka.

Instruments: ¹H and ¹³C-NMR was recorded using Ultra Shield 300 MHz 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 acrlro Erba 1106 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).

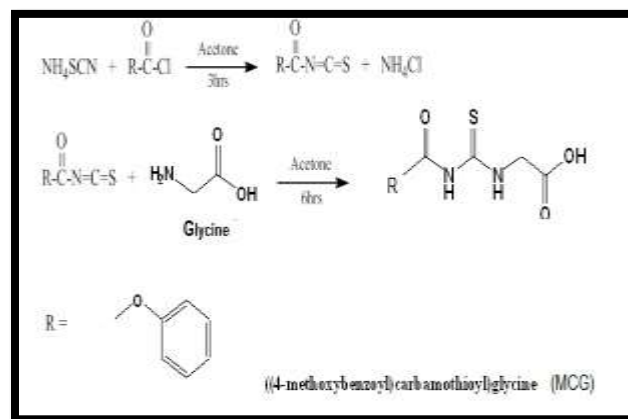
Preparation of (MCG):

1 Preparation of the (4 methoxy benzoyl isothiocyanate) [23]

Mixture of 4- methoxy benzoyl chloride (3.55 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

2 Preparation of (4 methoxy benzoyl) carbamothioyl) Glycine (MCG)

(1.95g, 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 (80%), (m.p =188-190) °C, C% found (49.22) while calculate (49.80), H% found (4.21) while calculate (4.50), N% found (9.94) while calculate e (10.60), S% found (11.96) while calculate (12.10).(Scheme(1)



Scheme 1: Preparation of Ligand (MCG)

Synthesis of metal complexes: (0.536g, 2mmole) of Ligand (MCG) was dissolved in 25ml of ethanol containing (0.12g, 2mmole) of KOH, then the solution of following metal salts MnCl₂·4H₂O (0.2g, 1mmole), CoCl₂·6H₂O (0.24g, 1mmole), NiCl₂·6H₂O (0.24g, 1mmole), CuCl₂·2H₂O (0.2g, 1mmole), ZnCl₂ (0.14g, 1mmole), CdCl₂·H₂O (0.2g, 1mmole), and HgCl₂ (0.3g, 1mmole) in ethanol, were added dropwise to the solution of (MCG·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 (MCG) and its metal complexes.

Table 1: Physical properties of (MCG) and its metal complexes

Compound	M.wt (gm/mol)	Color	M.p °C or dec.	M% Calculation (Found)	Molar condu. Ohm ⁻¹ Cm ² mol ⁻¹	μ _{eff} (B.M)
(MCG)	268	Deep Yellow	188-190	-	3.5	-
[Mn(MCG) ₂]	589	Brown	208- 210	9.00 (8.81)	6.35	5.96
[Co(MCG) ₂]	593	Deep Green	268-270	9.90 (9.30)	10.0	4.76
[Ni(MCG) ₂]	592	Brown	268	9.80 (8.68)	3.11	2.98
[Cu(MCG) ₂]	597	Green	228	10.60 (9.88)	6.1	1.71
[Zn(MCG) ₂]	599	Yellow	158	10.90 (10.12)	5.1	0
[Cd(MCG) ₂]	646.4	yellow	250-252	17.40 (16.80)	18	0
[Hg(MCG) ₂]	735	Yellow	213-215	27.0 (27.8)	3.5	0

Spectral studies

¹H and ¹³C-NMR spectra

1 ¹H-NMR spectrum for (MCG) in DMSO as solvent, Fig. (1) showed the following signals: singlet at δ (3.83)ppm for (3H,CH₃-O), singlet at δ(4.34) ppm for (2H, CH₂), multiplet at δ(2.48-2.49) ppm for DMSO, singlet at δ(7.83) ppm for (1H,NH sec.amine), multiplet at δ(7.84-7.95) ppm for (aromatic protons), singlet at δ(11.20) ppm for (1H, NH sec.amide), singlet at δ(7.97) ppm for (1H, COOH) [24] Table (2)

Table 2: (¹HNMR) Spectral data for ligand (MCG)

Compound	No of protons	(ppm)δ
Ligand (MCG)	S (3H,CH ₃ -O)	3.83
	S (2H,CH ₂)	4.34
	S (1H,NH aromatic)	(7.83)
	2d (4H,aromatic)	7.84-7.95
	S (1H,COOH)	7.97
	S (1H,NHsec.amide)	11.20

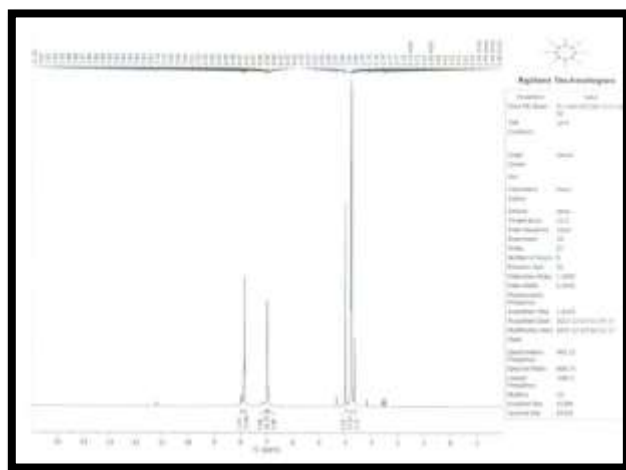


Fig 1: ¹HNMR spectrum of (MCG)

2 The ¹³C-NMR spectrum for (MCG) in DMSO, Fig. (2) showed the following signals: signals at δ (55.86)ppm for CH₃-O, signals at δ(52.15) ppm for (CH₂), multiplet at δ(39.35-40.35) ppm for DMSO,

signals at δ(125-164.61) ppm for aromatic carbons, signal at δ(166.33) ppm for (C=O sec.amide), signal at δ(170.25) ppm for (COOH), signal at δ(190.94) ppm for (C=S).

Table 3: ¹³CNMR Spectral data for ligand (MCG)

Compound	No of protons	δ(ppm)
Ligand (MCG)	s(CH ₂)	52.15
	s(CH ₃ -O)	55.86
	m (C -aromatic)	125-164.61
	s(C=Osec.amide)	166.33
	(C OOH) s	170.25
	s (C=S)	190.94

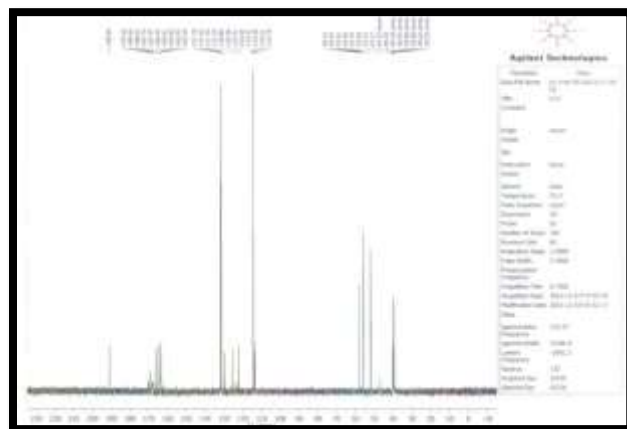


Fig 2: ¹³CNMR spectrum of Ligand (MCG) Infrared spectra

FT-IR spectrum of the free Ligand(MCG), Fig. (3), showed bands due to amido u(NH), u(C=O) and u(C=S) which absorbed at (3414) cm⁻¹, (1616) cm⁻¹, and (1257) cm⁻¹ respectively, while another absorption band appeared at (1685) cm⁻¹ could be explained as u(COO)_{asym} [25], [26] where the u(OCO)_{sym} was noticed at (1411) cm⁻¹. The FT-IR spectra of complexes exhibited marked difference between bands belonging to the stretching vibration of u(NH) of the amine group in the range between (3394 -3278) cm⁻¹ shifted lower frequencies by (20-136) cm⁻¹ suggesting the possibility of the coordination of (MCG) through the nitrogen atom at the amine group[27] Absorption assigned for

$\nu(\text{COO})_{\text{asym}}$ was noticed at the range (1558-1585) cm^{-1} shifted to higher frequencies by (100-127) cm^{-1} while the band caused by $\nu(\text{COO})_{\text{sym}}$ appeared between (1427-1370) cm^{-1} shifted to lower frequencies by (20-77) cm^{-1} which indicates to the coordination of the carboxylic group to the central ion [28].

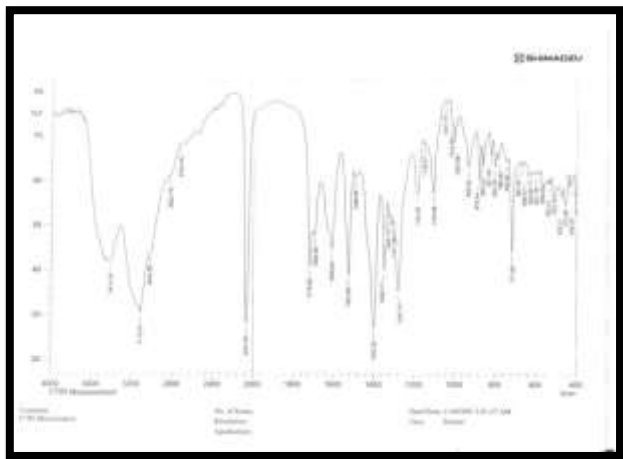


Fig 3: FT-IR spectrum of Ligand (MCG)

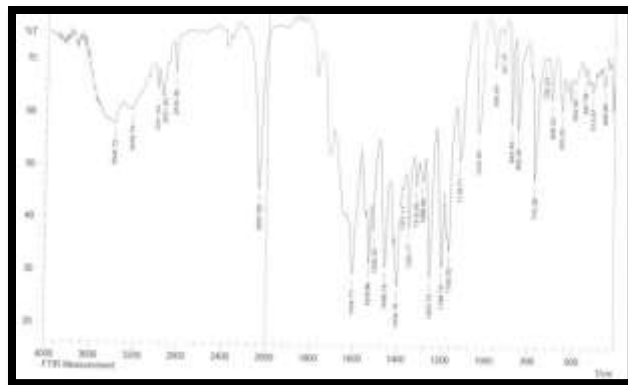


Fig 4: FT-IR spectrum of Complex $[\text{Co}(\text{MCG})_2]$

The stretching vibration band $\nu(\text{C}=\text{O})$ and $\nu(\text{C}=\text{S})$ carbonyl group either shows no change or very little in their frequencies (1604-1616) cm^{-1} and (1253-1257) cm^{-1} respectively there for indicating do not coordinate to the metal ion [29]. Metal-nitrogen and metal-oxygen bonds were confirmed by the presence of the stretching vibration of $\nu(\text{M}-\text{O})$ and $\nu(\text{M}-\text{N})$ around (447-532) cm^{-1} and (416-462) cm^{-1} respectively. Table (4) describes the important bands and assignment for free (MCG) and its complexes. The FTIR spectrum of (CO) complex showed in Fig. (3).

Table 4: Some FT-IR frequencies in (cm^{-1}) for (MCG) and its metal complexes

Complex	$\nu(\text{N}-\text{H})$	$\nu(\text{COO})_{\text{sym}}$	$\nu(\text{COO})_{\text{asym}}$	$\nu(\text{C}=\text{O})$	$\nu(\text{C}=\text{S})$	$\nu(\text{M}-\text{O})$	$\nu(\text{M}-\text{N})$
(MCG)	3414(s)	1411(m)	1685(s)	1616(m)	1257(s)	-	-
$[\text{Mn}(\text{MCG})_2]$	3391(m)	1438(w)	1558(m)	1616(m)	1257(m)	489(w)	462(w)
$[\text{Co}(\text{MCG})_2]$	3394(m)	1458(s)	1575(m)	1604(s)	1253(s)	513(w)	459(w)
$[\text{Ni}(\text{MCG})_2]$	3380(m)	1462(s)	1585(s)	1604(s)	1253(s)	466(w)	439(w)
$[\text{Cu}(\text{MCG})_2]$	3390(m)	1474(m)	1577(w)	1604(s)	1257(s)	501(w)	447(w)
$[\text{Zn}(\text{MCG})_2]$	3370(m)	1460(w)	1583(w)	1604(s)	1257(s)	470(w)	443(w)
$[\text{Hg}(\text{MCG})_2]$	3391(m)	1435(m)	1562(m)	1616(m)	1228(m)	532(w)	420(w)

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

Magnetic properties for the metal complexes: Magnetic moment (μ_{eff}) with regard to complexes related to $\text{Mn}^{+2}(\text{d}^5)$, also $\text{Co}^{+2}(\text{d}^7)$ have been indicated (5.96) B.M, also (4.76) B.M, that in anticipated spin-only values. Higher value related to μ_{eff} of $\text{Ni}^{+2}(\text{d}^8)$ complex (2.98) B.M because of orbital contributions [30]. Magnetic moment μ_{eff} related to $\text{Cu}^{+2}(\text{d}^9)$ complex indicated (1.71) B.M in expected value to one electron [31]. All data are indicated in the Table (1).

Electronic spectral: The UV-Visible spectra of Ligand (MCG) and its complexes recorded in Table (5), the solution of Ligand (MCG) in 10^{-3}M (DMSO) exhibited two peaks Fig. (5) at (38910) cm^{-1} and (17361) cm^{-1} which are attributed to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transition respectively [32].

The spectra of complexes

$[\text{Mn}(\text{MCG})_2]$ d^5 : The brown complex of Mn (II) shows bands at (38759) cm^{-1} and (14513) due to ligand field and the electronic transfer ${}^6\text{A}_1 \rightarrow {}^4\text{T}_2$ and another band at (13736) cm^{-1} which is caused by the electronic transfer ${}^6\text{A}_1 \rightarrow {}^4\text{T}_{1\text{G}}$ [33].

$[\text{Co}(\text{MCG})_2]$ d^7 : The spectrum of the deep green complex gave four bands at, (39062) cm^{-1} , (31250) cm^{-1} , (25575) cm^{-1} and (18018) cm^{-1} and (11173) cm^{-1} attributed to ligand field, (C.T), ${}^4\text{A}_2 \rightarrow {}^4\text{T}_{1(\text{P})}$, ${}^4\text{A}_2 \rightarrow {}^4\text{T}_{1(\text{F})}$ and ${}^4\text{A}_2 \rightarrow {}^4\text{T}_{2(\text{F})}$ respectively, and the Racah interelectronic repulsion parameter (B) was found to be (671.6) cm^{-1} , from the relation $\beta = B / B_0$, was found to be a equal (0.691), these parameter are accepted to Co(II) tetrahedral complex [34].

$[\text{Ni}(\text{MCG})_2]$ d^8 : The spectrum of Brown complex of Ni (II) has revealed the following electronic transfer ligand field, (C.T), mix with ${}^3\text{T}_{1(\text{F})} \rightarrow {}^3\text{T}_{1(\text{P})}$, ${}^3\text{T}_{1(\text{F})} \rightarrow {}^3\text{A}_{2(\text{F})}$ and ${}^3\text{T}_{1} \rightarrow {}^3\text{T}_{2(\text{F})}$, transition at (39215) cm^{-1} , (26385) cm^{-1} , (17331) cm^{-1} and (11607) cm^{-1} respectively, the (B) value found to be (593) cm^{-1} , while β was

equal to (0.57) these are the characteristics for tetrahedral complexes of Ni (II) [35].

$[\text{Cu}(\text{MCG})_2]$ d^9 : The spectrum of green complex of Cu(II) Fig. (6) show three bands at (38910) cm^{-1} , (13755) cm^{-1} and (11160) cm^{-1} caused to ligand field, (C.T), mix with ${}^2\text{B}_{1\text{g}} \rightarrow {}^2\text{A}_{1\text{g}}$ and ${}^2\text{B}_{1\text{g}} \rightarrow {}^2\text{B}_{2\text{g}}$ transition respectively [36].

The complexes of **$[\text{Zn}(\text{MCG})_2]$** , **$[\text{Cd}(\text{MCG})_2]$** and **$[\text{Hg}(\text{MCG})_2]$** shows only ligand field and charge transfer of (M→L) at (38610) cm^{-1} , (38910) cm^{-1} and (38610, 29411) cm^{-1} respectively [37]. All transition with their assignments are summarized in Table (5).

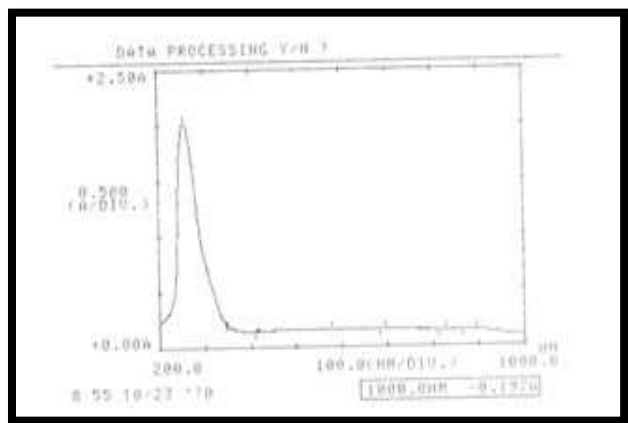


Fig 5: Electronic spectrum of ligand (MCG)

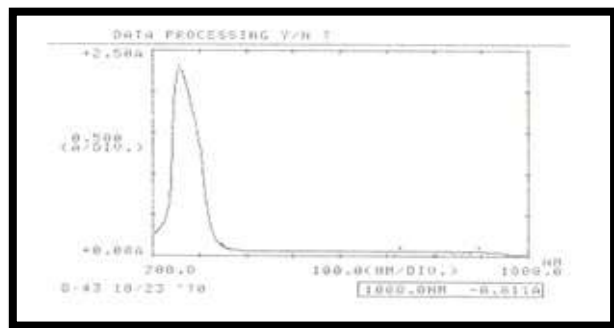
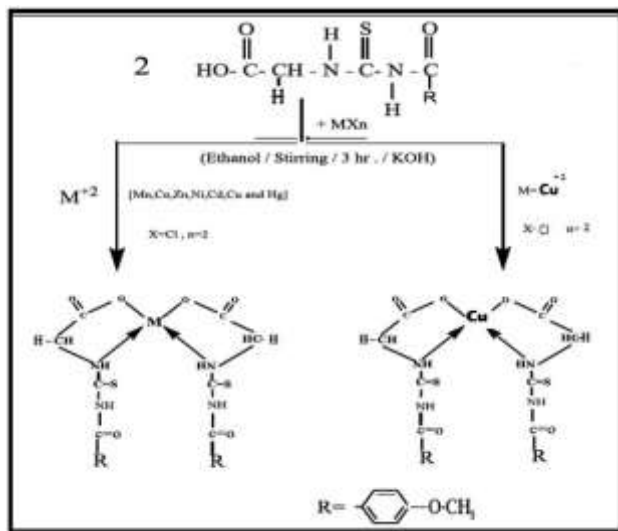
Fig 6: Electronic spectrum of Complex [Cu(MCG)₂]

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

الملاحظات	maxε L.mol ⁻¹ .cm ⁻¹	Wave number cm ⁻¹	ABC	λ _{max} (nm)	Complex	No
π → π*	2032	38910	2.032	257	MCG(المركب)	1
n → π*	163	17361	0.163	576		
L.F	2339	38759	2.339	258	[Mn(MCG) ₂]	2
⁶ A ₁ → ⁴ T ₂ (G)	143	14913	0.143	689		
⁶ A ₁ → ⁴ T ₁ (G)	140	13736	0.140	728		
L.F	2149	39062	2.149	256	[Co(MCG) ₂]	3
C.T	1080	31250	1.080	320		
⁴ A ₂ → ⁴ T _{1(p)}	720	25575	0.720	391		
⁴ A ₂ → ⁴ T _{1(f)}	98	18018	0.098	555		
⁴ A ₂ → ⁴ T ₂	61	11173	0.061	895		
L.F	2168	39215	2.168	255	[Ni(MCG) ₂]	4
³ T _{1(f)} → ³ T _{1(p)} C.T mix	112	26385	0.112	379		
³ T _{1(f)} → ³ A ₂	67	17331	0.067	577		
³ T _{1(f)} → ³ T _{2(f)}	61	11607	0.061	896		
L.F	2277	38910	2.277	257	[Cu(MCG) ₂]	5
² B _{1(g)} → ² A _{1(g)}	61	13755	0.061	727		
² B _{1(g)} → ² B _{2(g)}	59	11160	0.059	896		
L.F	2308	38610	2.308	259	[Zn(MCG) ₂]	6
L.F	2197	38910	2.197	257	[Cd(MCG) ₂]	7
L.F	2246	37453	2.246	259	[Hg(MCG) ₂]	8
C.T	453		0.453	340		

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

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(MCG)₂] where M²⁺ = (Mn, Co, Ni, Zn, Cd and Hg), Fig. (7) while Copper complex s has square planer

Fig 7: Suggested structure of complexes [M(MCG)₂]

CONCLUSION

The new ligand in the presented study was prepared through reaction from the 4-Methoxy benzoyl isothiocyanate with Glycine, ligand has been characterized through elemental micro analysis C.H.N.S., FTIR, UV Vis also ¹H,¹³C-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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