Synthesis and Characterization of [N-(4-Methoxybenzoyl amino)-thioxomethyl]Methionine acid (MbM) and It's Complexes with Some Divalent Transition Metals Ions (Mn , Co , Ni , Cu , Zn , Cd and Hg)

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Abstract

In this work , the ligand [N-(4-Methoxybenzoyl amino)-thioxomethyl] Methionine acid has been synthesized by the reaction of 4- Methoxybenzoyl isothiocyanate with methionine acid . The metal complexes were prepared through the reaction of metals chlorides of Co(II) , Ni(II), Cu(II), Zn(II) and Cd(II) in ethanol as solvent . The ligand (MbM) and its metal complexes have been characterized by elemental analysis (CHNS), IR, ^{1}H-$^{13}CNMR$ and UV-Vis spectra, magnetic susceptibility measurements, molar conductivity, melting points and atomic absorption. The metal-ligand ratio was determined by mole ratio method. The suggested structures for the Co(II), Ni(II), Cd(II) and Zn(II) complexes are tetrahedral geometry and the Cu(II) complex is square planer geometry.

Key Words: Methionine acid, 4-methoxy benzoyl isothiocyanate, complexes.

Theoretical

Methionine is one of the essential amino acids needed for good health but cannot be produced in the body, and so must be provided through our diet[1]. One of the important functions of methionine is its ability to be a supplier of sulfur and other compounds required by the body for normal metabolism and growth[2]. Sulfur is a key element and vital to our life. Without an adequate intake of sulfur, our body will not be able to make and utilize a number of antioxidant nutrients[3]. Methionine is also a methyl donor, capable of giving off a molecule with a single carbon atom with 3 tightly connected hydrogen atoms, called a methyl group which we need for a wide variety of chemical and metabolic reactions inside our body[4].

Methionine is capable of coordination through the – SCH_2 as well as through the – NH_2 and COO- groups and is potentially tridentate chelating ligand. On the other hand, since the (S) atom of this ether group (class b base) differs markedly in its donor properties from the N atoms of an amino group and the (O) atom of a carboxylate group (both class) bases methionine may not tend to coordinate with given metal ion as a tridentate chelating ligand (S,N, and O donor atoms)[5]. More likely methionine could be expected to act as a bidentate chelating ligand and use different pairs of different metal atoms[6].

Methionine with molecular formula, $C_5H_{11}NO_2S$ is one of the two sulfur containing amino acid, cysteine being the other. Methionine helps to initiate translation of messenger RNA by being the first amino acid incorporated into the (N) terminal position of all proteins. It is considered as an essential amino acid for normal metabolism, growth and maintenance of body tissue. It is used as nutritional supplement and act as antioxidant in biological system[7-9].

Some metal complexes of DL–methionine were prepared in aqueous medium and characterized by different physico-chemical methods[10]. Methionine forms 1:2 complexes with metal, M(II). The general empirical formula of the complexes is proposed as $[(C_5H_{10}NO_2S)_2M]$; where M^{+2} = Co, Ni, Cu, Zn, Cd and Hg. All the complexes are extremely stable in light and air and optically inactive. These transition metals are essential trace elements and used as nutritional supplement. They act as cofactors in various enzyme systems i.e. as metalloenzymes or as enzymatic activators[7-10]. Cd(II) and Hg(II) are toxic elements, that methionine is a biological chelating agent may lower the degree of toxicity for the formation of chelate with toxic metals[11-13].

The aim of this work is the preparation of some new transition metal complexes of [N-(4-methoxybenzoyl amino)-thioxomethyl] methionine acid (MbM).

Experimental

Chemicals

All chemicals were supplied from Al-Drich, Fluka and BDH.

Materials

(4-Methoxybenzoyl chloride), (Methionine acid), Manganese chloride tetrahydrate ($MnCl_2.4H_2O$), Cobalt chloride hexahydrate ($CoCl_2.6H_2O$), Nickel chloride 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 ($HgCl_2$).

Instruments

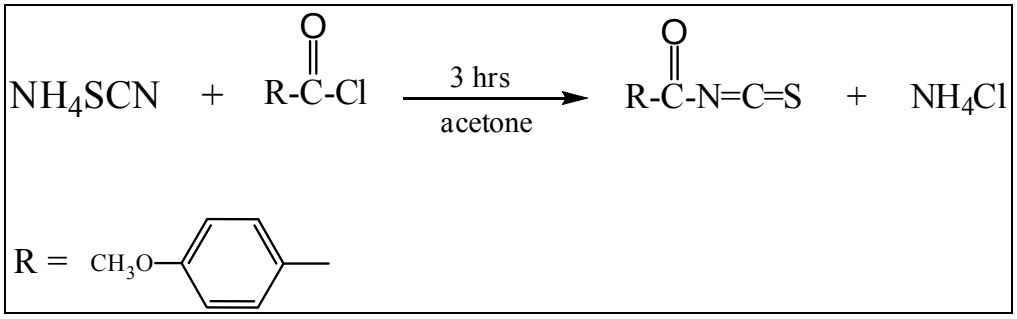
The UV-Vis spectra have been recorded in the range of (200-1000) nm using (Shimadzu U.V-165PCS spectrophotometer). Infrared spectra have been recorded in the range (400-4000) cm^{-1} using KBr disk for the ligand and its complexes using (Shimadzu FT-IR 8400S spectrophotometer).

The ^{1}H-NMR and ^{13}C-NMR spectra was recorded on Bruker model Ultra shield 300 MHz NMR Switzerland at Al-Albyt University of Jordon. DMSO was used as solvent and TMS as internal reference. Elemental analysis were recorded on instrument type EA-99.mth. The metal content was determined by using Shimadzu corporation model AA-6300 atomic absorption. Molar conductivity measurements were obtained by using electrolytic conductivity measuring set model Cond 3110 SET1 in DMSO solvent in concentration (10^{-3}M) at 25^{o}C. Magnetic susceptibility measurements were obtained at room temperature applying method using Balance Magnetic Susceptibility Model MSB-MKI.

Synthesis of the ligand [N-(4-Methoxybenzoyl amino)-thioxomethyl] methionine acid (MbM)

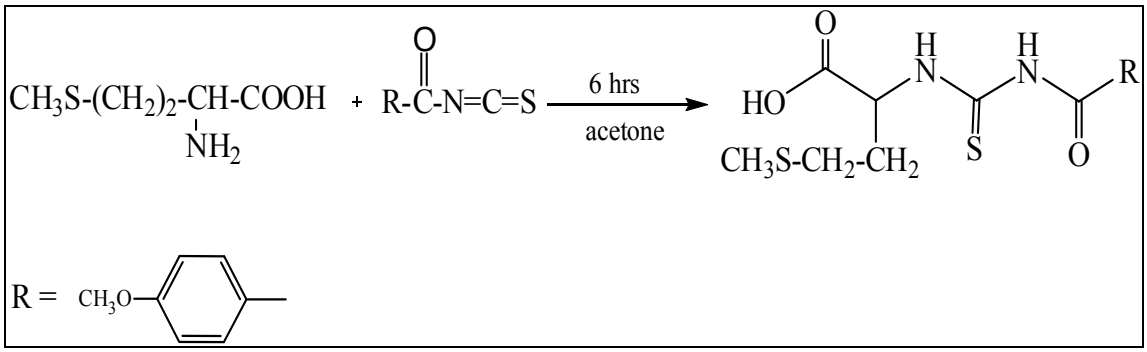
1- Preparation of the (4-Methoxybenzoyl isothiocyanate)

Mixture of 4-methoxy benzoyl chloride (3.55ml, 1mmol) and ammonium thiocyanate (2g, 1mmol) in (25 ml) of acetone was stirred under refluxed for 3 hours and then filtered, the filtrate was used for further reaction[14].



2- Preparation of [N-(Methoxybenzoyl amino) thioxomethyl] methionine acid (MbM)

(3.42g, 1mmol) of methionine acid 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 (84%) , (m.p =194-196) ˚C, %C found (49.06) while calculate (49.10), %H found (5.30) while calculate (5.29), %N found (9.97) while calculate (8.68), %S found (18.30) while calculate (18.72).



Scheme No. (1)

Synthesis of the Metal Complexes

The ligand (MbM) (0.002mol, 0.68gm) was dissolved in 25 ml ethanol in a 100 ml round-bottom flask containing (0.12g, 0.002mol) of KOH. A solution of (0.001mol, 0.2gm, 0.001mol, 0.24gm, 0.001mol, 0.24gm, 0.001mol, 0.2gm, 0.001mol, 0.14gm, 0.001mol, 0.2gm, 0.001mol, 0.3gm, 0.001mol) of the hydrated metal chloride Mn(II), Co(II), Ni(II), Cu(II), Zn(II), Cd(II) and Hg(II) respectively in 20 ml ethanol was added dropwise, with continuous stirring at room temperature for 3hr.

The resulting precipitates were filtered off, washed with ethanol, dried and recrystallized from ethanol. Physical properties were given in Table (1).

Results and Discussion

The ligand and its complexes are soluble in organic solvents, such as DMSO and DMF 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 the ligand and it's complexes.

Spectral Studies

^{1}H-NMR spectrum of ligand (MbM)

The proton nuclear magnetic resonance spectrum for the ligand (MbM) was carried out using (DMSO) as a solvent and the following peaks were detected, Figure (1):
single peak at δ(0.9 – 2.06) ppm for (3H , CH_3S), quartet peak at δ(2.19 - 2.33) ppm for (2H , CH_2), triplet peaks at δ(2.35 - 2.44) ppm for (2H , CH_2S)**,** single peak at δ(2.50) ppm for DMSO, single peak at δ(3.26 - 3.51) ppm for (3H , O-CH_3), quartet peak at δ(4.69 - 4.71) ppm for (1H , CHCOOH), pairs of doublet at δ(6.62 – 7.67) ppm for 4H proton aromatic ring, single peaks at δ(10.9) ppm for (1H , NH sec. amide), single peak at δ(11.10) ppm for (1H , COOH).

^{13}C-NMR spectrum of ligand (MbM)

The carbon nuclear magnetic resonance spectrum for the ligand (MbM) was carried out using (DMSO) as a solvent and the following peaks were detected, Figure (2):
signal at δ(17.00) ppm for (CH_3S), signals at δ(29.44 – 31.82) ppm for (CH_2S) group, signals at δ(38.62 – 40.29) ppm for DMSO, signals at δ (43.49) ppm for (CH_2), signals at δ(49.99 – 58.34) ppm for (CH), signals at δ(92.87 – 161.56) ppm for aromatic carbons, signals at δ(163.00– 167.49) ppm for (C=O sec. amide), singles at δ(170.78 – 174.71) ppm for (COOH), singles at δ(180.53 – 182.17) ppm for (C=S).

Infrared Spectra

FT-IR spectrum of the free ligand (MbM), Figure (3), showed bands due to amido υ(NH), υ(C=O) and υ(C=S) which absorbed at (3317) cm^{-1}, (1666) cm^{-1} and (1257) cm^{-1} respectively, while another absorption band appeared at (1728) cm^{-1}could be explained as $\upsilon(COO)_{asym}$[15,16], where the $\upsilon(OCO)_{sym}$ was noticed at (1404) cm^{-1}.

The FT-IR Spectra of Complexes

These spectra exhibited marked difference between bands belonging to the stretching vibration of υ(NH) of the amine group in the range between (3209-3301) cm^{-1} shifted lower frequencies by (108-16) cm^{-1} suggesting of the possibility of the coordination of ligand through the nitrogen atom at the amine group[17]. Absorption assigned for $\upsilon(COO)_{asym}$ was noticed at the range (1604-1620) cm^{-1} shifted to lower frequencies by (108-124) cm^{-1} while the band caused by $\upsilon(COO)_{sym}$ appeared between (1496-1542) cm^{-1} shifted to higher frequencies by (92-138) cm^{-1} which indicates to the coordination of the carboxylic group to the central ion[18]. The stretching vibration band υ(C=O) and υ(C=S) carbonyl group either show no change or very little in their frequencies (1650-1666) cm^{-1} and (1257) cm^{-1} respectively there for indicating do not coordinate to the metal ion[19]. Metal- nitrogen and metal-oxygen bonds were confirmed by the presence of the stretching vibration of υ(M-O) and υ(M-N) around (456-516) $cm^{-1}$and (401-450) cm^{-1} respectively, Table (2) describes the important bands and assignment for free ligand (MbM) and it's complexes. FT-IR spectrum of the Mn complex, Figure (4).

Molar Conductivity Measurements

The molar conductance values of the synthesized complexes were measured in DMSO solvent and concentration 10^{-3}M. The spectral data of molar conductance are shown in Table (1) and in the range (8-20 $ohm^{-1}cm^2$ $mole^{-1}$). These results inducted to the complexes are non-ionic[20,21].

Electronic Spectra

The UV-Visible of the ligand (MbM) and its complexes recorded in Table (3). The solution of the ligand (MbM) in 10^{-3}M (DMSO) exhibited one peak, Figure (5) at (37755) cm^{-1} which are attributed to ***π-π**** transition[22].

The Spectra of Complexes

-[Mn(MbM)$_2$] d^5: the pale yellow complex of Mn(II) shows bands at (37878) cm^{-1} due to charge transfer and another bands at (17730) cm^{-1} and (10893) cm^{-1} which are caused by the electronic transfer $^6A_1 \rightarrow {}^4T_2(D)$ and $^6A_1 \rightarrow {}^4T_1(D)$ respectively[23].

-[Co(MbM)$_2$] d^7: the spectrum of the pale green complex gave four bands at (37735) cm^{-1}, (18903) cm^{-1},(18115) cm^{-1} and (10121) cm^{-1}attributed to (C.T),$^4A_2 \rightarrow {}^4T_1(P)$, $^4A_2 \rightarrow {}^4T_1(F)$ and $^4A_2 \rightarrow {}^4T_2(F)$ transitions respectively. The rach interelectronic repulsion parameter (B^-) was found to be (443.67) cm^{-1}, from the relation $\beta = B^- / B_0$ was found to be equal (0.457), these parameter are accepted to Co(II) tetrahedral complex[24].

-[Ni(MbM)$_2$] d^8: the spectrum of pale green complex of Ni(II), Figure (6), has revealed the following electronic transfer (C.T), $^3T_1(F) \rightarrow {}^3T_1(P)$, $^3T_1(F) \rightarrow {}^3A_2(F)$, and $^3T_1 \rightarrow {}^3T_2(F)$ transition at (36363) cm^{-1}, (20833) cm^{-1}, (17006) cm^{-1} and (10152) cm^{-1} respectively.

The (B^-) value found to be (492.2) cm^{-1}, while β was equal to (0.470) these are the characteristics for tetrahedral complexes of Ni(II)[25].

-[Cu (MbM)$_2$] d^9: the spectrum of pale green complex of Cu(II) show three bands at (37735) cm^{-1}, (14836) cm^{-1} and (11737) cm^{-1} caused to (C.T) , $^2B_{1g} \rightarrow {}^2A_{1g}$ and $^2B_{1g} \rightarrow {}^2B_{2g}$ transition respectively[26].

- [Zn(MbM)2], [Cd(MbM)2] and [Hg(MbM)2] show only charge transfer of (M L) in range (35842-37174) cm-1[27]. All transition with their assignments are summarized in Table (3).
Study of complexes formation in solution

Complexes of ligand (MbM) with metal ions were studied in solution using ethanol as solvent in order to determine [M/L] ratio in complexes follow molar method[28]. A series of solution were prepared having A constant concentration (10-3M) of metal ion and ligand.

The [M/L] ratio is determined from the relationship between the absorption of the absorbed light and the mole ratio of [M/L]. The results of complexes in ethanol suggest that the metal to ligand ratio was [1:2] for all complexes which were similar to that obtained from solid state study .

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(MbM)2], where M+2= (Mn, Co, Ni, Zn, Cd and Hg), Figure (7) while copper complexes [Cu(MbM)2] have square planer.

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Table No. (1): Some physical properties of the ligand (MbM) and its complexes

Compound	M.wt (gm/mole)	Color	M.P(C°)	M% Calculation (found)	Molar Condu. $Ohm^{-1}cm^2mol^{-1}$ in DMSO	μ_{eff}(B.M)
(MbM)	342.43	Brawn	94-95	-	8	-
$Mn(MbM)_2$	737.79	Pale yellow	220-222	7.60 (6.95)	20	5.86
$Co(MbM)_2$	741.78	Dark green	250-252	8.11 (7.77)	17	5.13
$Ni(MbM)_2$	741.54	Pale green	270	8.08 (7.65)	19	3.12
$Cu(MbM)_2$	746.40	Pale green	190-192	8.69 (8.10)	20	1.78
$Zn(MbM)_2$	748.23	Pale yellow	250	8.92 (8.32)	12	0
$Cd(MbM)_2$	795.26	Pale yellow	270(dec)	14.41 (15.10)	9	0
$Hg(MbM)_2$	883.44	Pale yellow	185 (dec)	23.10 (22.85)	12	0

Table No. (2): Some IR frequencies in (cm^{-1}) for the ligand (MbM) and its metal complexes

Compound	υ(N—H)	$\upsilon(COO)_{sym}$	$\upsilon(COO)_{asym}$	υ(C=O)	υ(C=S)	υ(M-O)	υ(M—N)
LH=Ligand	3317 (m)	1404 (s)	1728(s)	1666 (s)	1257(s)	—	—
$[Mn(MbM)_2]$	3232 (b)	1496 (s)	1604 (s)	1666 (w)	1257 (s)	509 (m)	401 (w)
$[Co(MbM)_2]$	3209 (b)	1496(m)	1604 (s)	1666(w)	1257 (s)	516 (m)	410 (w)
$[Ni(MbM)_2]$	3240 (b)	1504(m)	1604 (s)	1650 (w)	1257 (s)	516 (w)	401 (w)
$[Cu(MbM)_2]$	3301 (b)	1504(m)	1604 (s)	1666 (m)	1257 (s)	516 (m)	401 (w)
$[Zn(MbM)_2]$	3290 (m)	1504(m)	1620 (m)	1650 (w)	1257 (s)	516 (w)	450 (w)
$[Cd(MbM)_2]$	3224 (m)	1504(s)	1604 (s)	1666 (m)	1257 (s)	509 (s)	430 (m)
$[Hg(MbM)_2]$	3255 (b)	1542(s)	1604 (s)	1666 (m)	1257 (s)	456 (m)	401 (m)

HL=ligand (MbM) b=browed W=weak S=strong m= medium

Table No. (3): The peaks, electronic transitions and structure geometries of the ligand (MbM) and its complexes

Comp.	λ_{max} (nm)	υ^-(cm^{-1})	ABC	ε_{max}	Transitions
ligand (MbM)	265	37735	1.46	1460	π-π*
$Mn(MbM)_2$	264 564 918	37878 17730 10893	1.129 0.010 0.021	1129 10 21	C.T $^6A_1 \rightarrow {}^4T_2(D)$ $^6A_1 \rightarrow {}^4T_2(D)$
$Co(MbM)_2$	265 529 552 988	37735 18903 18115 10121	1.230 0.194 0.190 0.028	1230 1940 0.190 28	C.T $^4A_2(F) \rightarrow {}^4T_1(P)$ $^4A_2(F) \rightarrow {}^4T_1(F)$ $^4A_2(F) \rightarrow {}^4T_2(F)$
$Ni(MbM)_2$	275 480 588 985	36363 20833 17006 10152	1.280 0.056 0.025 0.066	1280 56 25 66	C.T $^3T_1(F) \rightarrow {}^3T_1(P)$ $^3T_1(F) \rightarrow {}^3A_2(F)$ $^3T_1(F) \rightarrow {}^3T_2(F)$
$Cu(MbM)_2$	265 674 852	37735 14836 11737	1.271 0.134 0.047	1271 134 47	C.T $^2B_1g \rightarrow {}^2A_1g$ $^2B_1g \rightarrow {}^2B_2g$
$Zn(MbM)_2$	279	35842	1.644	1644	C.T
$Cd(MbM)_2$	279	35842	0.938	938	C.T
$Hg(MbM)_2$	269	37174	0.505	505	C.T

C.T = Charge transfer

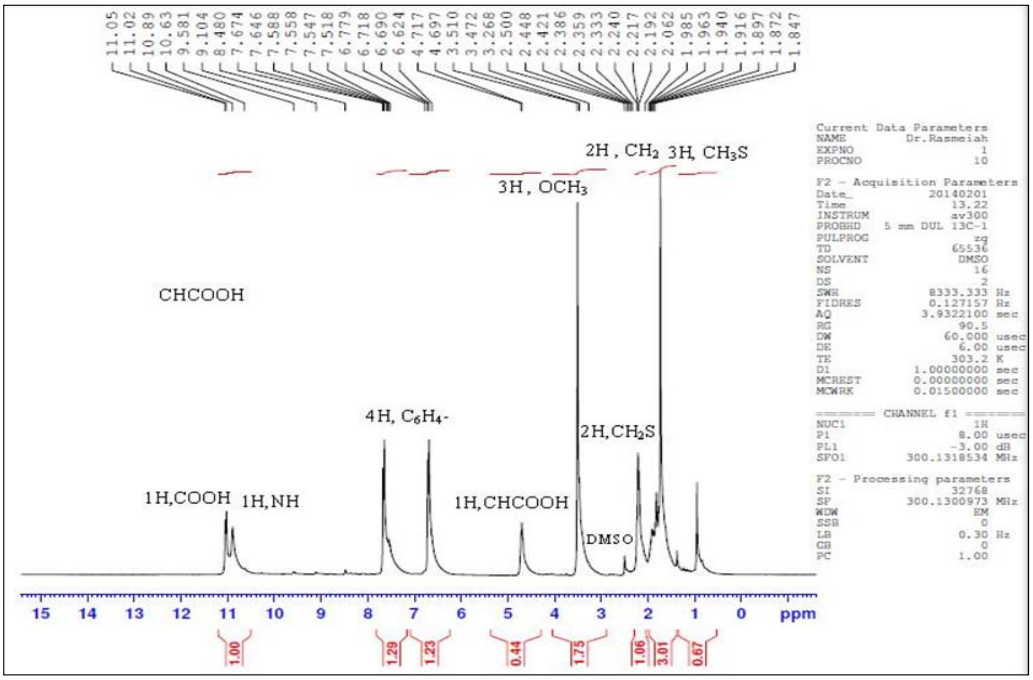

Figure No. (1): ^{1}H-NMR spectrum of ligand (MbM)

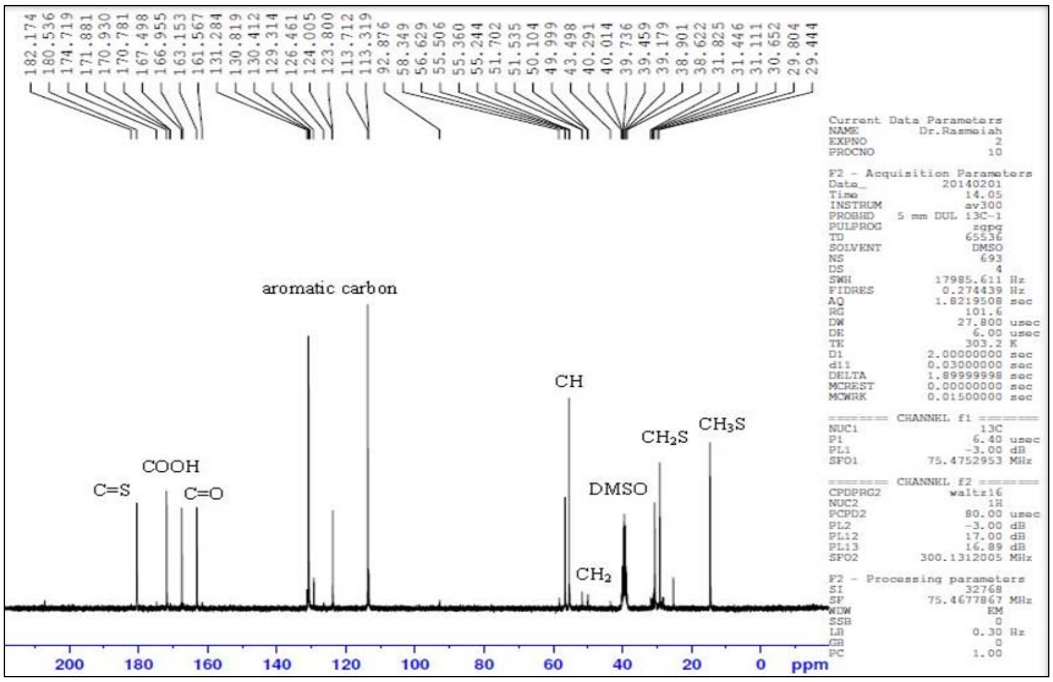

Figure No. (2) : ^{13}C-NMR spectrum of ligand (MbM)

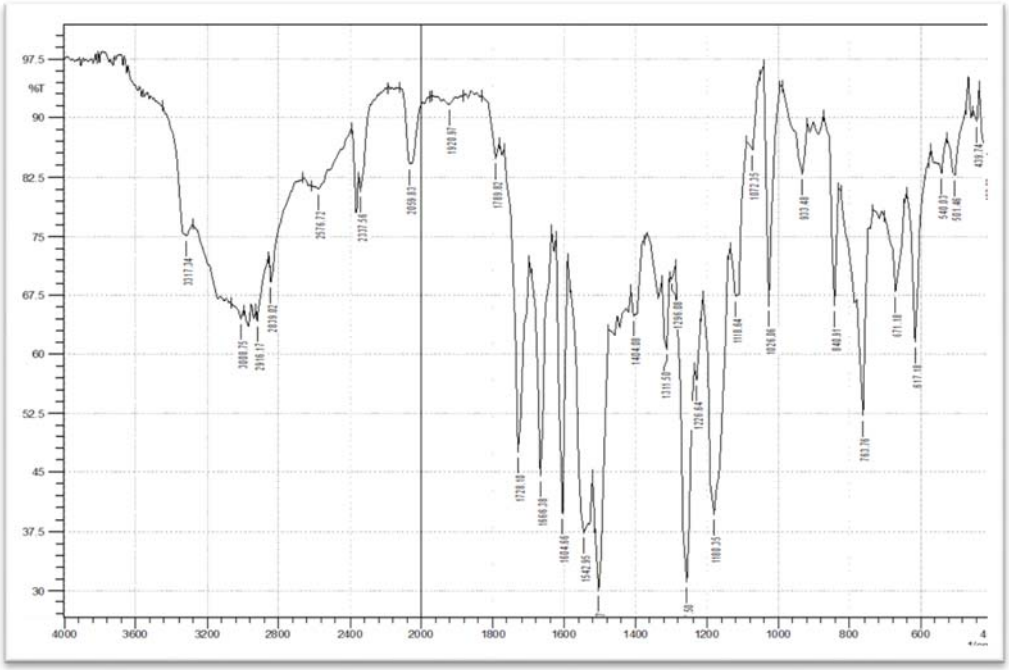

Figure No. (3) : Infrared spectrum for ligand (MbM)

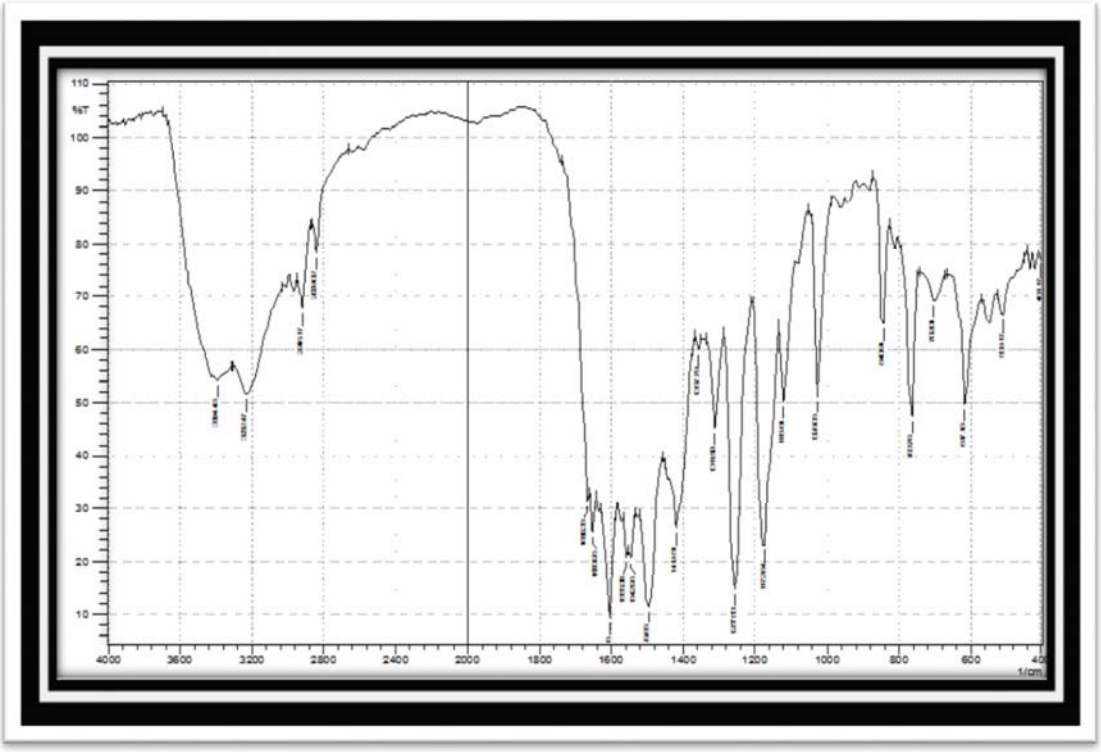

Figure No. (4): Infrared spectrum of [Mn(MbM)$_2$] complex

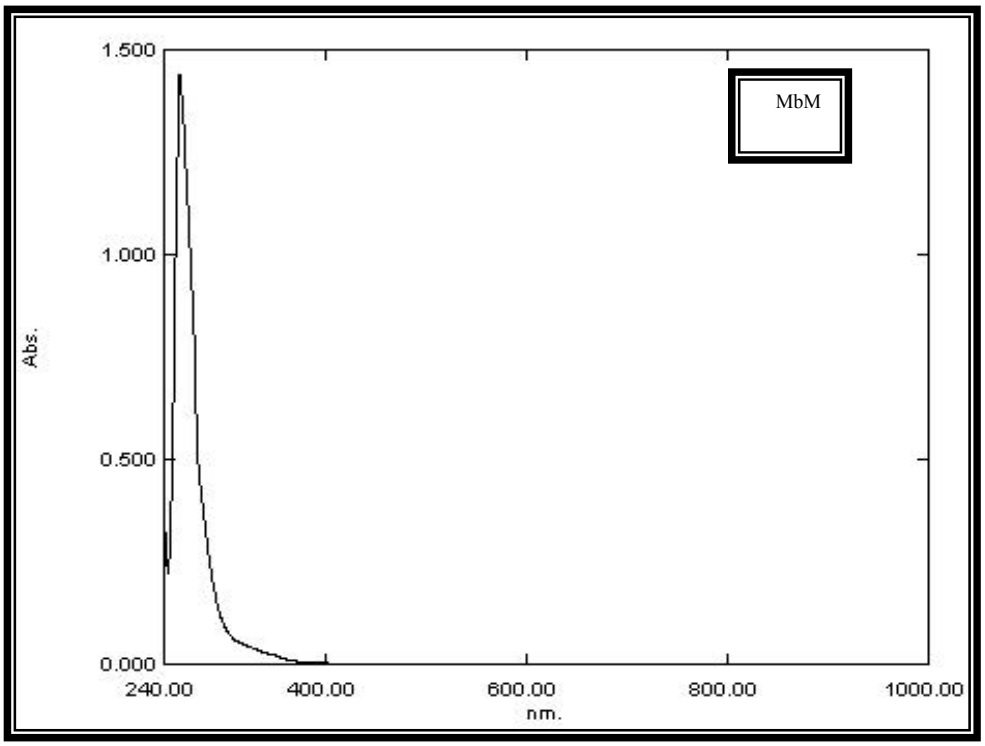

Figure No. (5): U.V spectrum of ligand (MbM)

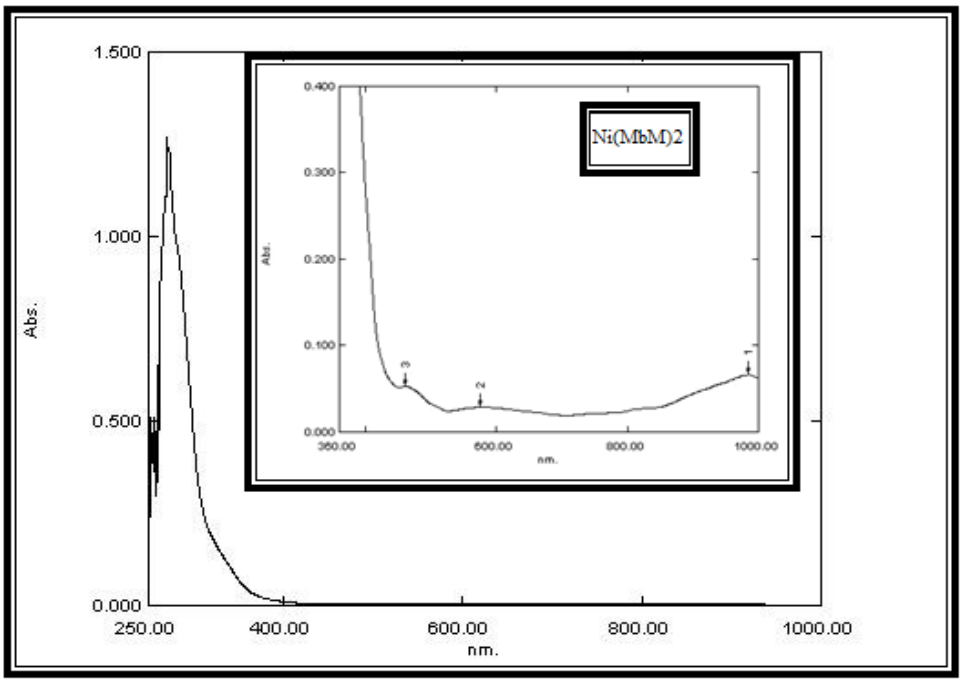

Figure No. (6): U.V spectrum of [Ni(MbM)$_2$] complex

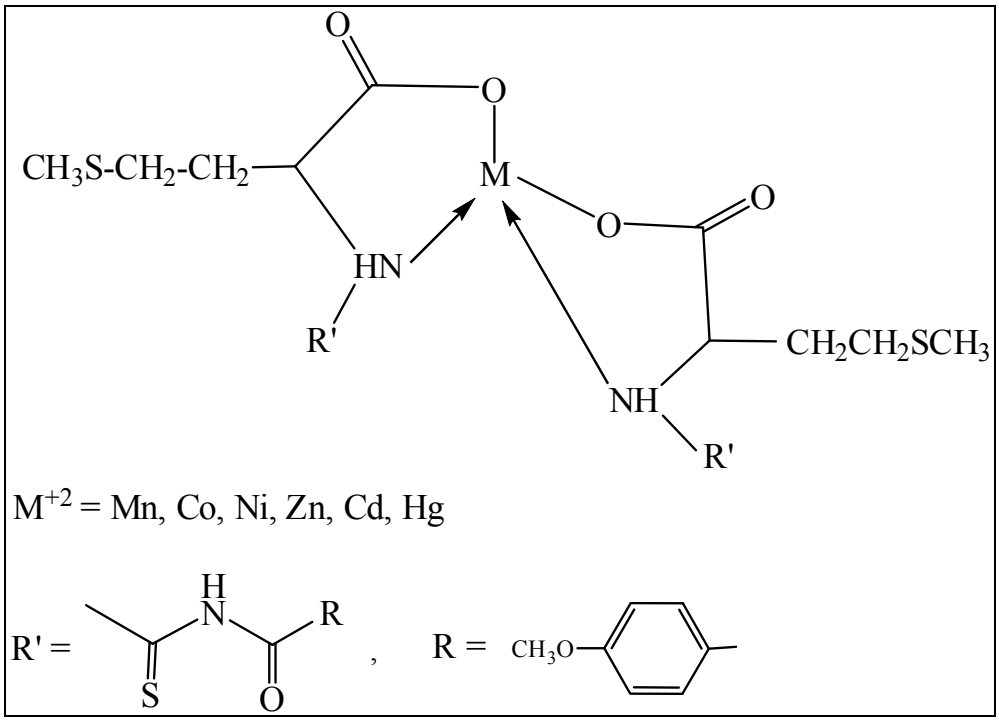

Figure No. (7): The proposed chemical structure formula of the complexes

تحضير وتشخيص بعض المعقدات الفلزية الجديدة للأيونات ثنائية التكافؤ (Mn، Co، Ni، Cu، Zn، Cd، Hg) مع [N- (4- ميثوكسي بنزويل امينو- ثايواوكسو مثيل)] الميثيونين

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الخلاصة

حضر الليكاند الجديد (MbM) [N- (4- ميثوكسي بنزويل امينو- ثايواوكسو مثيل)] الميثيونين و ذلك من مفاعلة (4- ميثوكسي بنزويل ايزوثايوسيانات) مع حامض الميثيونين وبنسبة (1:1) وشخص بوساطة التحليل الدقيق للعناصر(CHNS) والأشعة تحت الحمراء والأشعة فوق البنفسجية - المرئية وطيف الرنين النووي المغناطيسي, كما حضرت وشخصت معقدات بعض ايونات العناصر الانتقالية الثنائية التكافؤ (Mn , Co , Ni , Cu , Zn , Cd , Hg) مع الليكاند (MbM) باستخدام الأشعة تحت الحمراء والأشعة فوق البنفسجية – المرئية والتوصيلية المولارية والحساسية المغناطيسية والامتصاص الذري وتحليل النسبة المولية واستنتج من الدراسات والتشخيصات أن المعقدات لها شكل رباعي السطوح حول الايون الفلزي مع الليكاند (MbM) ثنائي السن ماعدا معقد النحاس الذي أعطى الشكل المربع المستوي.

الكلمات المفتاحية : حامض الميثيونين, 4- كلوروبنزويل ايزوثايوسيانيت, معقدات.