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Synthesis and Characterization of Some Metal Complexes of [4-Methoxy-N-(pyrimidine-2-ylcarbamothioyl)benzamide]

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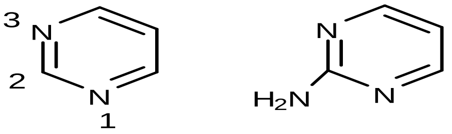
Abstract:

A new ligand [4-Methoxy -N-(pyrimidine-2-ylcarbamothioyl) benzamide] (MPB) was synthesized by reaction of (4-Methoxyibenzoyl isothiocyanate)with (2-aminopyri-midine). The Ligand was characterized by elemental micro analysis (C.H.N.S),(FT-IR) (UV- Vis) and (^{1}H ,^{13}CNMR)spectra. Some transition metals complexes of this ligand were prepared and characterized by (FT-IR, UV-Vis) spectra conductivity measurements magnetic susceptibility and atomic absorption. From the obtained results the molecular formula of all complexes was suggested to be [$M(MPB)_2Cl_2$] (M^{+2} =Cu, Mn, Co ,Ni ,Zn ,Cd and Hg),the proposed geometrical structure for all complexes was an octahedral.

Keywords: 2-Amino Pyrimidine, 4-Methoxy benzoyl isothiocyanate, complexes.

Introduction:

Nitrogen containing heterocycles act an important part in medicinal chemistry and also contribute to the society by helping as in different life processes[1]. Heterocyclic compounds are torrential in nature and are of great significance to life because their structural subunits exist in many natural products such as vitamins, hormones, antibiotics etc. A practical method for the synthesis of such compounds is of great interest in synthetic organic chemistry. Pyrimidine is a six-member heterocyclic compound that contains two nitrogen atoms at positions 1 and 3. The structure of the pyrimidine ring is similar to benzene and pyridine The key role pyrimidines play in cellular processes has made them valuable leads for drug discovery. Pyrimidine derivatives are known to be biologically active compounds and substituted Pyrimidines have shown a wide range of biological activities like anti tubercular antibacterial, antioxidant, anti-inflammatory activity[2]. The chemistry of pyrimidines and their derivatives have been studied since past centuryidue to their close pharma cological association with diverse pharmacological properties. Pyrimidine Scheme(1-a) was first isolated in 1899. Though pyrimidine itself does not exist in nature but substituted Pyrimidines moiety are found as part of more complex systems and are widely distributed. Pyrimidines[3]. are considered to be important not only because they are an integral part of the genetic material DNA and RNA as nucleotides and nucleosides but they also impart numerous biological activities such as bactericides, fungicides, viricides, insecticides and meticides. They have also found applications in agricultural and industrial chemicals.



[a] [b]

Scheme 1. structures of pyrimidine and 2-Aminopyrimidine

2-Aminopyrimidine Scheme(1-b) is an interesting structural element present in several marketed drugs, and its derivatives possess divers biological activities, e.g.
Antipsychotic, cardoprotective, and antimalarial [4]. Schiff bases derived from 2-aminopyrimidine have been used as antibacterial [5], Antitumor agents[6]. In this work, a new ligand was prepared by the reaction of 2-Aminopyrimidine with N-2,4-Dihydroxy benzaldehyde. Also it's complexes were prepared, (where M^{2+} =Cu, Pd, Zn, Co, Ni and M^{3+}= In,Ir)[7].

Materials and Methods:

Chemicals: The chemicals used in this work were all of reagent grade by BDH, Merck and Fluka.

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Materials: (4-Methoxybenzoylchloride), (2-aminopyrimidine), Manganese chloride tetrahydrate ($MnCl_2.4H_2O$), Cobalt chloride hexahydrate ($CoCl_2.6H_2O$),Nickel chloride hexahydrate ($NiCl_2.6H_2O$), Copper chloride hydrate ($CuCl_2.2H_2O$), Zinc chloride ($ZnCl_2$), Cadmium chloride hydrate ($CdCl_2.H_2O$) and Mercury chloride ($HgCl_2$).

Instruments

^1H and ^{13}C–NMR spectra were recorded using Ultra Shield 300 MH_z Switzerl and at University of Al al-Bayt, Jordan. Melting points were recorded by using Stuart- melting point apparatus. FT-IR spectra were recorded as CsI disc using 3800 Shimadzu in the range of (4000 -200)cm^-. Electronic spectra were obtained using UV- 160 Shimadzu spectro photometer at 25℃ for10^{-3}M solution DMSO with 1.000 ± 0.001 cm matched quartz cell. Molar Conductivity was measured at 25℃ for 10^{-3}M solution of DMSO using Philips P_W. Digital .micro elemental analysis (C.H.N.S) were performed using Acrlo Erba 1106 elemental analyzer. Magnetic susceptibility measurements were obtained magnetic susceptibility balance model MSB-MKI. Metal contents of the complexes were determined by atomic absorption technique using Shimadzu (AA680G).

Preparation of ligand (MPB)

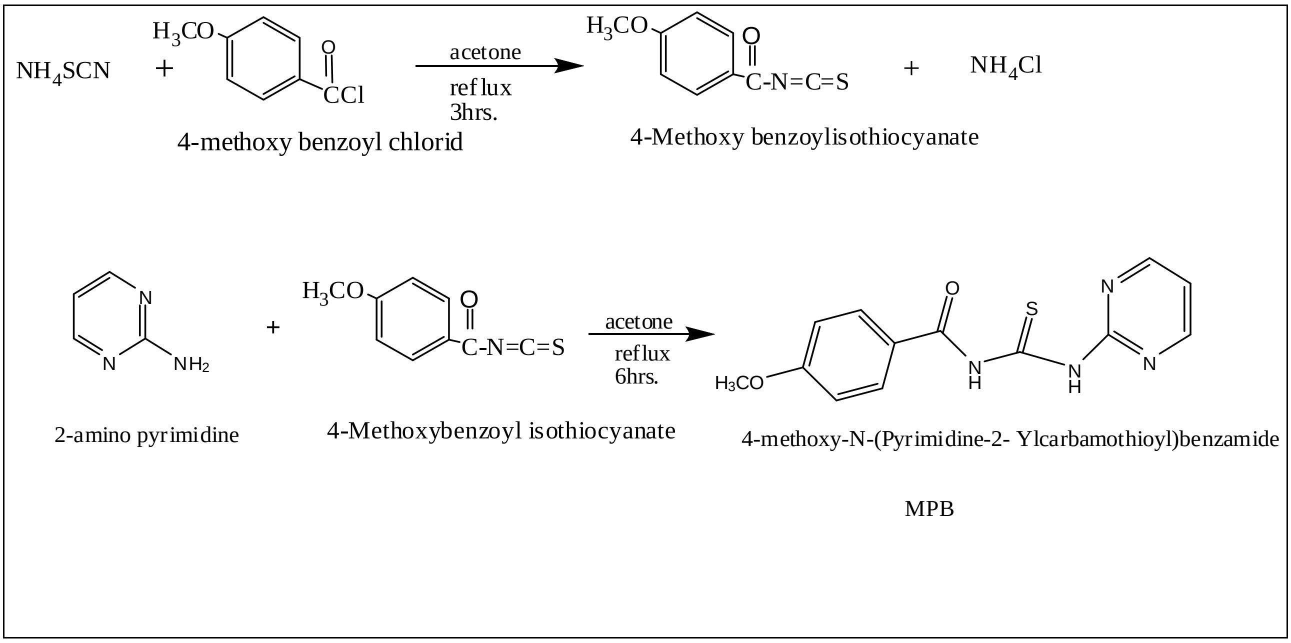
The ligand was prepared by two steps (scheme-2)

(A)- Preparation of the (4- Methoxybenzoyl isothiocyanate

Mixture of 4-Methoxy benzoyl chloride (3.51ml,26 mmol) and ammonium thiocyanate (2g, 26mmol) in 25 ml acetone was stirred under reflux for 3hrs then filtered, the filtrate was used for further reaction. [8]

(B)- Preparation of [4-Methoxy -N-(pyrimidin-2-ylcarbamothioyl)benzamide] (MPB)

A solution of (2.47g,26mmol) 2-Aminopyrimidinein 20 mlacetone was rapidly added to 4- methoxy benzoyl isothiocyanate and maintaining reflux. After refluxing for 6hrs the resulting solid was collected, washed with acetone, and recrystallized from ethanol, yield (79%), (m.p=170-172)˚C, C% found (53.99 calc. (54.11), H% found(5.36) calc. (5.29),N %found(19.63) calc.(19.43) ,S% found (11.8) calc.(11.12).



Scheme 2. The synthesis of route ligand [MPB]

Synthesis of the ligand complexes

Synthesis of the [Zn(MPB)$_2$Cl$_2$] complex

A solution of (0.136 g, 1mmol) $ZnCl_2$ in 10 ml ethanol was added to a solution of (0.576g,2mmol) (MPB)in10 ml ethanol. The mixture was stirred under refluxed for 6 hrs at room temperature, the brown solid product was collected by filtration, washed with (1:1) mixture of water: ethanol, and dried in an oven at 50˚C.

Synthesis of [Cu (MPB)$_2$Cl$_2$], [Mn (MPB)$_2$Cl$_2$], [Co(MPB)$_2$Cl$_2$], [Ni(MPB)$_2$ Cl$_2$], [Cd(MPB)$_2$ Cl$_2$]and [Hg (MPB)$_2$Cl$_2$], complexes .

A similar method to that mentioned for preparation of [Zn(MPB)$_2$Cl$_2$] complex was used to prepare the complexes of [Cu^{+2}, Mn^{+2} , Co^{+2}, Ni^{+2}, Cd^{+2} and Hg^{+2}]ions with (MPB) ligand**.** Some physical properties of the prepared complexes are shown in Table(1).

Table 1. Some physical properties of the ligand(MPB) and its Complexes

Molecular formula	Mwt gm/mol^{-1}	Color	M.P Or dec. (°C)	M% Calculated (Found)	Molar Cond. Ohm^{-1}cm^{2}mol^{-} in DMSO	μeff (B.M)
($C_{13}H_{12}O_2N_4S$) (MPB)	288.32	Orange	170-172	-	-	-
[Mn (MPB)$_2$CI$_2$]	702.546	Orange	285-287	7.82 (7.64)	17.2	5.87
[Co(MPB)$_2$CI$_2$]	706.476	Green	305(dec)	8.34 (8.19)	7.71	4.61
[Ni(MP)$_2$CI$_2$]	706.256	Green	280-282	8.31 (8.42)	14.2	3.01
[Cu(MPB)$_2$CI$_2$]	710.546	Brown	310 (dec)	8.86 (8.64)	12.0	1.77
[Zn(MPB)$_2$CI$_2$]	710.916	Orange	288-290	8.91 (8.79)	13.4	0
[Cd(MPB)$_2$CI$_2$]	759.946	Orange	300 (dec)	14.79 (14.68)	18	0
[Hg(MPB)$_2$CI$_2$]	848.146	Orange	297 (dec)	23.63 (23.45)	8.5	0

dec. = decomposition

Results and Discussion:

Ligand (MPB)

- The FT-IR spectrum of the free ligand (MPB), Figure(1) shows bands at(1681)cm^{-1} and (1253)cm^{-1} dueto,υC=O(amidic) and υ(C=S) respectively. While another absorption band at (3379)cm^{-1} could be explained as υ N-H [9,10]. Spectral data of the free ligand are listed in Table (2).

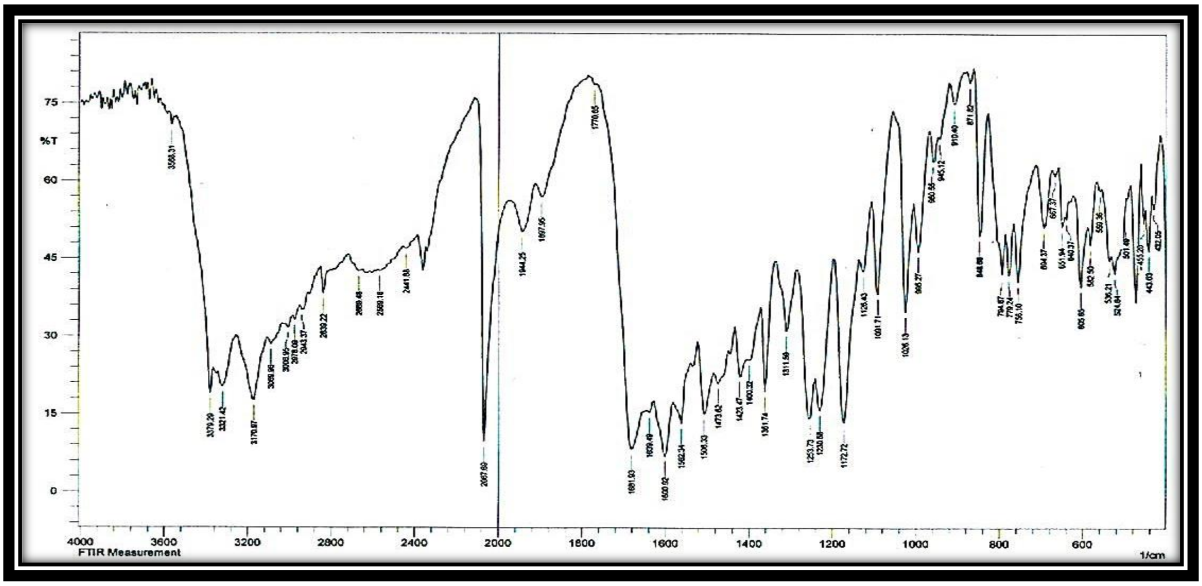


Figure 1. FT-IR Spectrum of the Ligand(MPB).

Table 2. The characteristic bands of FT-IR Spectra of ligand (MPB)and its metal complexes.

Compound	υ (N-H)	υ (C=O) Amide	υ (C=S)	υ (M-O)	υ (M-S)	υ (M-Cl)
Ligand(MPB)	3379 (s)	1681 (s)	1253 (s)	-	-	-
[Mn(MPB)$_2$Cl$_2$]	3375 (s)	1649 (s)	1234 (s)	474(s)	329 (m)	250(m)
[Co(MPB)$_2$Cl$_2$]	3378 (m)	1647 (s)	1228 (m)	428 (s)	314 (w)	273(w)
[Ni(MPB)$_2$Cl$_2$]	3373 (m)	1658 (m)	1200 (s)	470 (s)	329(w)	229 (w)
[Cu(MPB)$_2$Cl$_2$]	3377 (m)	1612 (s)	1224 (m)	464 (m)	322 (w)	297 (w)
[Zn(MPB)$_2$Cl$_2$]	3375 (m)	1604 (m)	1234 (s)	432 (m)	306 (m)	260 (m)
[Cd(MPB)$_2$Cl$_2$]	3363 (s)	1662 (s)	1201 (s)	482 (s)	337 (m)	264 (w)
[Hg(MPB)$_2$Cl$_2$]	3380 (s)	1666 (s)	1226 (s)	449 (s)	327 (m)	262 (m)

s=strong w=weak m=medium b=broad UV-Vis spectra

-The UV-Vis spectrum of the free ligand (MPB). Figure.(2)exhibits a high intense absorption peak at (34722)cm^{-1}which may be attributed to electronic transition type $\pi \longrightarrow \pi^*$ [11] The data of electronic spectrum of the free ligand (MPB) is listed in Table (3).

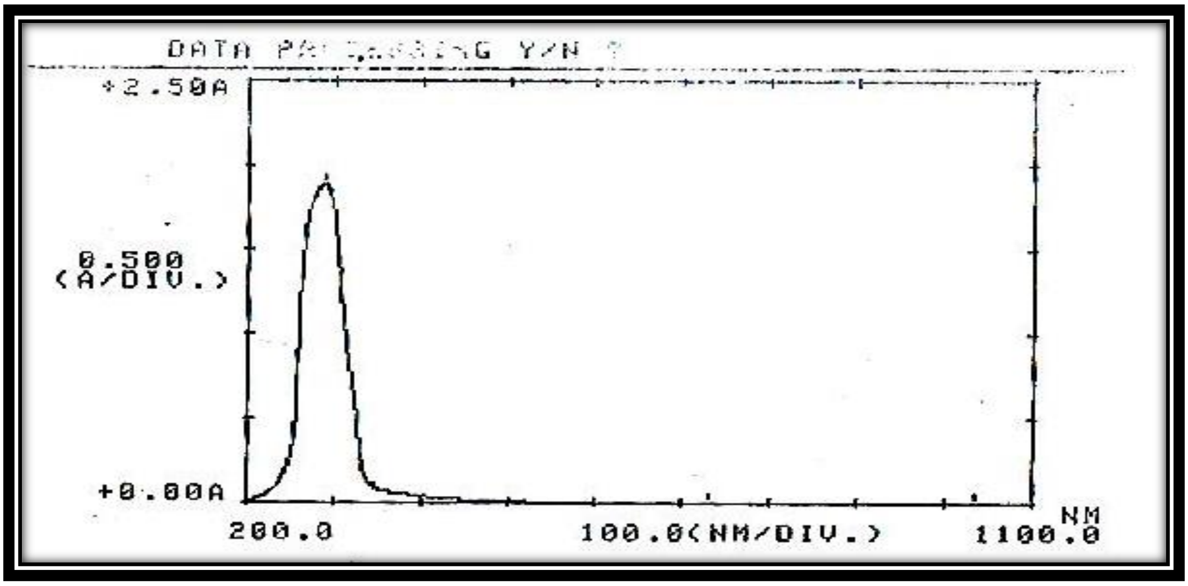


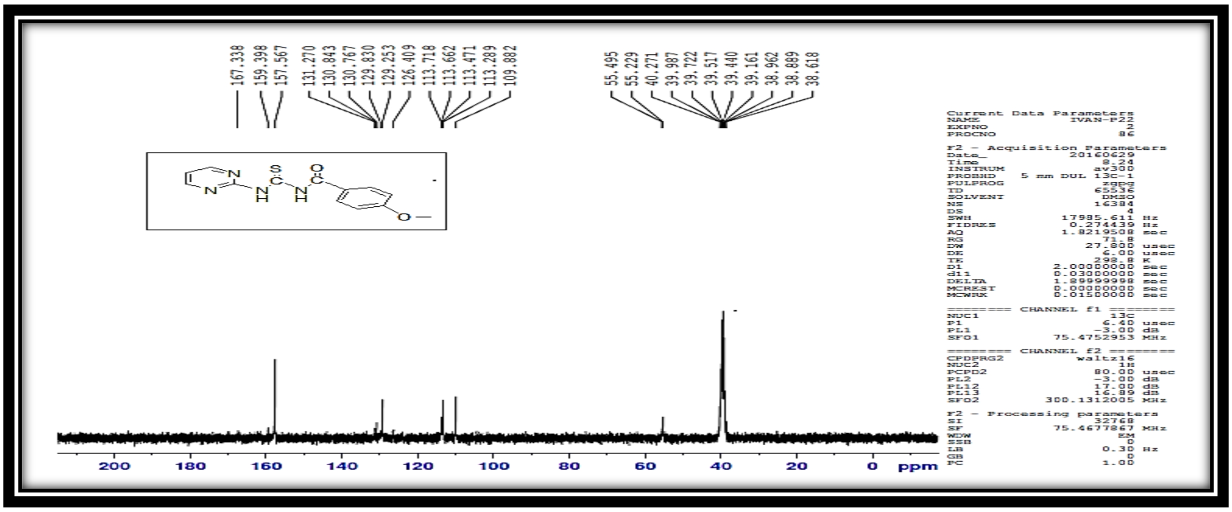
Figure 2. Electronic spectrum of ligand (MPB).

Table 3. Electronic spectral data of ligand (MPB) and its complexes in DMSO

Compounds	λ(nm)	$\bar{\upsilon}$(cm^{-1})	ABC	ε_{max} molar^{-1}cm^{-1}	Transitions
(Ligand MPB)	288	34722	1.884	1884	$\pi \longrightarrow \pi^*$
$[Mn(MPB)_2Cl_2]$	275	36363	1.872	1872	L.F
	784	12755	0.020	20	$^6A_1g \longrightarrow {}^4T_2g_{(G)}$
	866	11547	0.018	18	$^6A_1g \longrightarrow {}^4T_1g_{(G)}$
$[Co\ (MPB)_2CI_2]$	276	36231	2.167	2167	L.F
	392	25510	0.095	95	$^4T_1g_{(F)} \longrightarrow {}^4T_1g_{(P)}$
	517	19342	0.018	18	$^4T_1g_{(F)} \longrightarrow {}^4A_2g$
	864	11574	0.015	15	$^4T_1g_{(F)} \longrightarrow {}^4T_2g_{(F)}$
$[Ni(MPB)_2CI_2]$	282	45460	2.416	2416	L.F
	368	27173	0.272	272	C.T overlap with $^3A_2g_{(F)} \longrightarrow {}^3T_1g_{(P)}$
	554	18015	0.020	20	$^3A_2g_{(F)} \longrightarrow {}^3T_1g_{(F)}$
	890	11235	0,015	15	$^3A_2g_{(F)} \longrightarrow {}^3T_2g_{(F)}$
$[Cu(MPB)_2CI_2]$	273	36630	1.925	1925	L.F
	864	11574	0.025	25	$^2Eg \longrightarrow 2T_2g$
$[Zn\ (MPB)_2CI_2]$	291	34364	2.457	2457	L.F
$[Cd(MPB)_2CI_2]$	271	36900	1.481	1481	L.F
$[Hg\ (MPB)_2CI_2]$	274	36496	1.978	1978	L.F

L.F = ligand field, C.T = charge transfer

The ^{1}H-NMR spectrum of free ligand (MPB), Figure.(3) was recorded in DMSO-d6 solvent showed the following signals: singlet peak at δ(2.5) ppm or DMSO ,singlet Peak at (3.79) ppm for (3H,OCH_3), multiplet peak at δ(6.73-8.73) ppm for aromatic protons), singlet peak at δ(9.90) ppm refers to (1H,NH sec . amine), signals peak at δ(11.00) ppm, for (1H,NHsec. amide).



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PROCNO                  86

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PULPROG               zgpg
TD                   65536
SOLVENT               DMSO
NS                   16384
DS                       4
SWH              17985.611 Hz
FIDRES            0.274439 Hz
AQ               1.8219508 sec
RG                    71.8
DW                  27.800 usec
DE                    6.00 usec
TE                   298.8 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                    6.40 usec
PL1                  -3.00 dB
SFO1            75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
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PL13                 16.89 dB
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F2 - Processing parameters
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GB                       0
PC                    1.00
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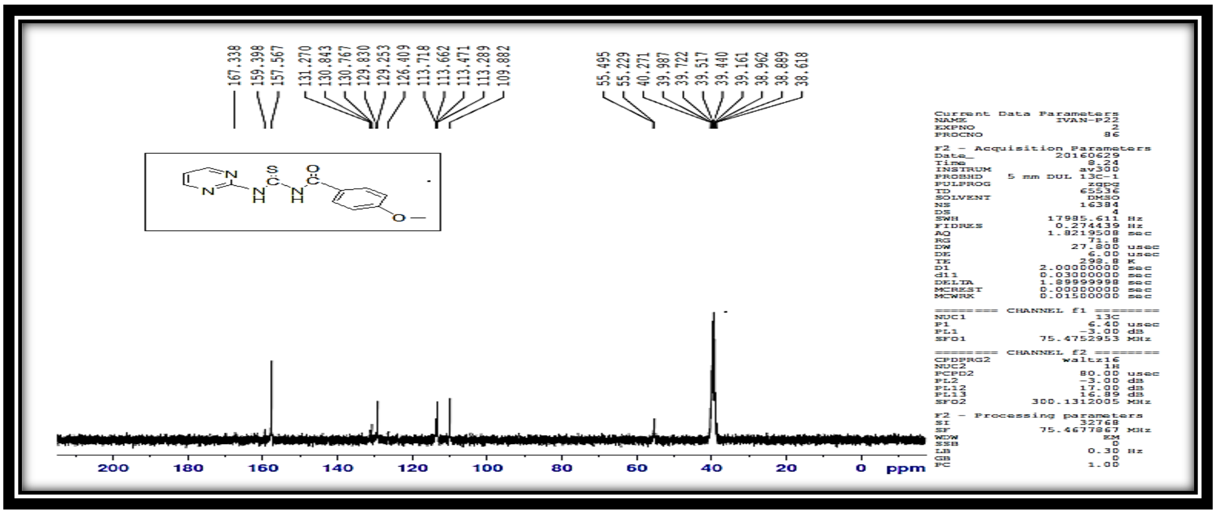
Figure 3. ^{1}H-NMR spectrum of ligand(MPB).

- ^{13}CNMR spectrum of the free ligand (MPB) in DMSO-d^6, Fig.(4) showed for the following signals: singlet at δ(38.61-40.27) ppm for DMSO, signal at δ(55.22-55.49) ppm for (OCH$_3$), signal at δ(109.88-157.56) ppm for aromatic carbons, signal at δ(159.39) ppm for (C=Osec.amid), signal atδ (167.33) ppm for(C=S)[9,12]



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SOLVENT               DMSO
NS                   16384
DS                       4
SWH              17985.611 Hz
FIDRES            0.274439 Hz
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RG                    71.8
DW                  27.800 usec
DE                    6.00 usec
TE                   298.8 K
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d11             0.03000000 sec
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PL1                  -3.00 dB
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======== CHANNEL f2 ========
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NUC2                    1H
PCPD2                80.00 usec
PL2                  -3.00 dB
PL12                 17.00 dB
PL13                 16.89 dB
SFO2           300.1312005 MHz

F2 - Processing parameters
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WDW                     EM
SSB                      0
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GB                       0
PC                    1.00
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Figure 4. ^{13}C -NMR spectrum of ligand(MPB).

Complexes of the ligand(MPB)

The solid complexes are soluble in some common solvents such as dimethyl form amide, dimethyl sulphoxide and relatively thermally stable. The molar conductivity values of all complexes in DMSO solvent in10^{-3}M at 25˚C(Table-1) indicated non-electrolyte. The atomic absorption measurements for all complexes gave approximated values when compared with theoretical values, Table(1) includes the physical properties for the ligand and its complexes.

FT-IR Spectra of complexes

These spectra exhibited marked differences between bands, Figure(5). belonging to the stretching vibration of υ(C=O amido)in the range between (1666-1604) cm^{-1}shifted to lower frequencies by(77-15)cm^{-1} suggesting the possibility of the coordination of ligand through the oxygen atom at the carbonyl group [14], while the band caused by υ(C=S) appeared between (1234-1200)cm^{-1} shifted to lower frequencies by (75-19)cm^{-1}which indicates the coordination of ligand through the sulfur atom at the thion group to the centralt ion [13-15]. The stretching vibration band υ(N-H) either shows no change or very little in their frequencies (3380-3363)cm^{-1} indicating to non coordination between the metal ion on the metal ligand. Metal-oxygen and metal-sulfur bonds were confirmed by the presence of the stretching tremor of υ(M-O),υ(M-S) and υ(M-Cl) around (482-424)cm^{-1}, (337-306) cm^{-1} and (229-297)cm^{-1} respectively. the spectra of complexes [14].Table (2) describe the important bands and assignment for free ligand (MPB) and its complexes.

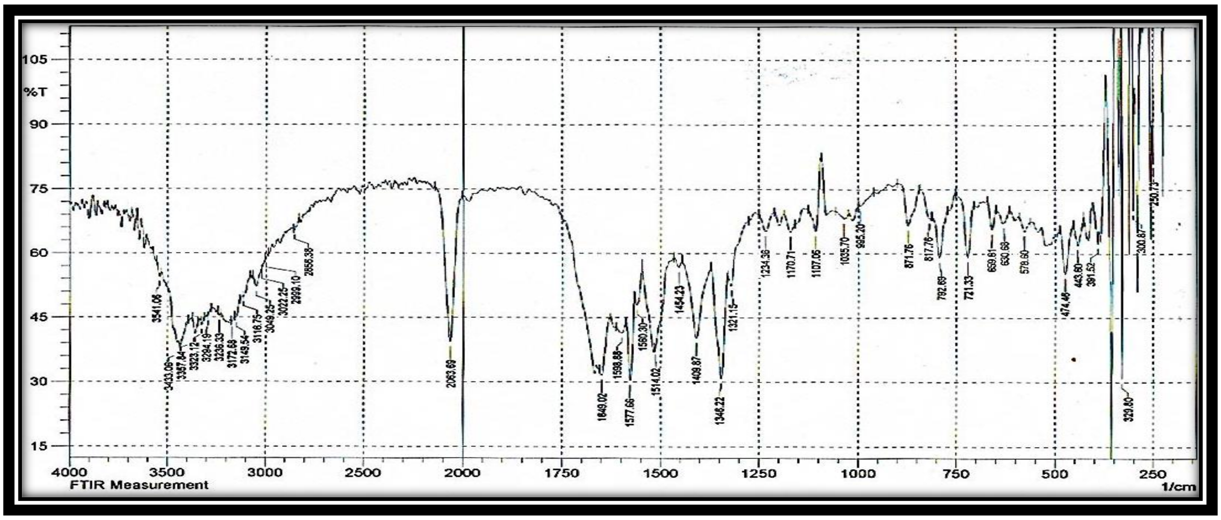


Figure 5. FT-IR spectrum of [Mn(MPB)$_2$Cl$_2$]

Magnetic moment

The values of measured magnetic susceptibility and effective magnetic moment (μeff) for the Mn (II), Co(II) ,Ni (II) and Cu(II) complexes are shown in Table(1). Mn(II), Co(II), Ni (II) and Cu (II) complexes exhibit μeff (5.87, 4.61, 3.01,1.77) B.M respectively, which reflect normal values for high spin octahedral complexes.[16]

Electronic spectra for complexes

- [Mn(MPB)$_2$Cl$_2$] d^5

The Orange complex of Mn (II), Figure (6),shows band at(36363)cm^{-1} due to (L.F) and other bands at (12755)cm^{-1} and (11547)cm^{-1}which are attribted to (d-d) the electronic transition type $^6A_1g \longrightarrow {}^4T_2g_{(G)}and^6A_1g \longrightarrow {}^4T_1g_{(G)}$ respectively, suggesting an octahedral geometry around Mn (II) ion [17].

-[Co(MPB)$_2$Cl$_2$] d^7

The spectrum of the Green complex gave four bands at (36231)cm^{-1} (25510)cm^{-1} (19342)cm^{-1} and (11574)cm^{-1} attributed to (L.F), $^4T_1g_{(F)} \longrightarrow {}^4T_1g_{(P)}$, $^4T_1g_{(F)} \longrightarrow {}^4A_2g_{(F)}$ and $^4T_1g_{(F)} \longrightarrow {}^4T_2g_{(F)}$ respectively and the rach inter electronic repulsion parameter (B$^-$) was found to be (675)cm^{-1}, from the relation β=B$^-$ / B^0, was found to be equal (0.695); these parameter are accepted to Co(II) octahedral comp-lex[18].

-[Ni(MPB)$_2$Cl$_2$] d^8

The spectrum of Green complex of Ni(II) has exhibited four (L.F), $^3A_2g \longrightarrow {}^3T_1g_{(P)}$, $^3A_2g \longrightarrow {}^3T_1g_{(F)}$, and $^3A_2g \longrightarrow {}^3T_2g_{(F)}$, which refer to electronic transition bands at(35460) cm^{-1}, (27173)cm^{-1}, (18050)cm^{-1}, (11235)cm^{-1} respectively. The (B$^-$)value is found to be (767.8) cm^{-1},while β was equal to(0.73). These are the characteristics for octahedral complexes of Ni(II)[19].

-[Cu (MPB)$_2$Cl$_2$] d^9

The spectrum of Brown complex of Cu(II) shows two bands at (36630) cm^{-1} (11574)cm^{-1} refers to (L.F),$^2Eg \longrightarrow {}^2T_2g_{(F)}$, electronic transition respectively which was in good agreement for distorted octahedral complex for Cu(II) ion[20].

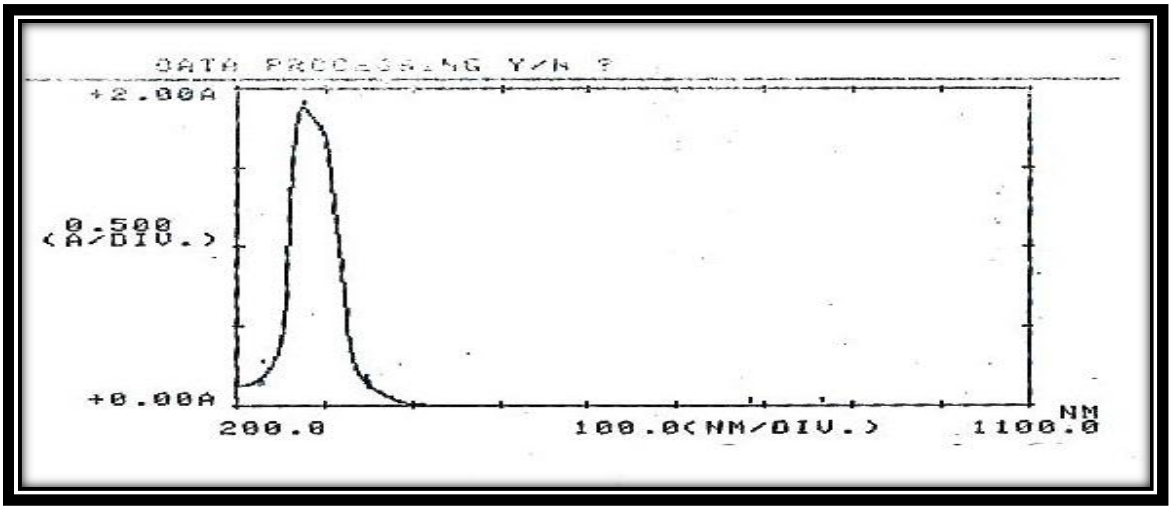
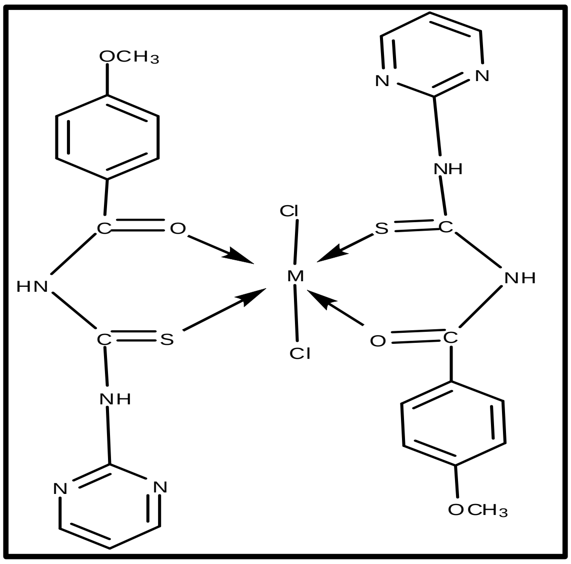


Figure 6. Electronic spectrum of [Mn(MPB)$_2$Cl$_2$]

-The complexes of $[Zn(MPB)_2Cl_2]$, $[Cd(MPB)_2Cl_2]$ and $[Hg(MPB)_2Cl_2]$

The spectrum of these complexes displayed one peak at range(271-291)nm (36900-34364)cm^{-1}which attributed to L- F [21]. with their assignments are summarized in Table(3).Suggested structures for complexes on the basis of molar conductivity , magnetic moment ,spectroscopic studies (FT-IR, UV-Vis and atomic absorption) and ^1H-^{13}CNMR for ligand (MPB) only the ligand (MPB) behaves as bi dentate on coordination with Mn(II), Co(II), Ni(II), Cu(II), Zn(II),Cd(II) and Hg(II) ions via oxygen atom of (C=O) amido group and sulfur atom of (C=S) group, suggesting octahedral geometry around metal ions for all the prepared complexes.



M = Mn (II), Co(II), Ni(II) , Zn(II), Cd(II) and Hg(II)

Figure 7. The proposed chemical structure formula of the complexes

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تحضير وتشخيص بعض المعقدات الفلزية مع [4- ميثوكسي- N -(بيرميدين- 2-يل كاربامو ثايويل) بنزاميد]

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الخلاصة:

حضر الليكاند الجديد4- ميثوكسي- N- (بيرميدين- 2-يل كارباموثايويل) بنزاميد(MPB) من مفاعله (4-ميثوكسي بنزويل ايزوثايوسيانات) مع 2-امينوبيرميدين وبنسبة (1:1) وشخص بوساطة التشخيص الدقيق للعناصر(C.H.N.S) والأشعة تحت الحمراء-(FT-IR)والأشعة فوق لبنفسجية- المرئية(UV-Vis) وطيف الرنين النووي المغناطيسي (^{1}H,^{13}CNMR) ، كما حضرت وشخصت معقدات بعض ايونات العناصر الانتقالية الثنائية التكافؤ(Hg, Cd, Zn, Cu, Ni, Co, Mn)مع الليكاند (MPB) وشخصت المعقدات المحضرة باستعمال الأشعة تحت الحمراء والأشعة فوق البنفسجية - المرئية والتوصيلية المولارية والحساسية المغناطيسية والامتصاص الذري وتم الاستنتاج من الدراسات التشخيصيه وإن المعقدات لها شكل ثماني السطوح حول الايون الفلزي مع الليكاند (MPB) ثنائي السن.

الكلمات المفتاحية: 2-امينوبيرميدين ،4-ميثوكسي بنزويل ايزوثايوسيانات، معقدات.