Synthesis and Characterization of Some Metal Complexes of [4-chloro-N-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-ylcarbamothioyl) benzamide]

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Abstract

A new ligand [4-chloro-N-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-ylcarb amothioyl) benzamide] (CAP) was synthesized by reaction of P-ChloroBenzoyl isothio cyanate with 4-aminoantipyrine,The ligand was characterized by micro elemental analysis C.H.N.S.,FT-IR,UV-Vis and ^{1}H-$^{13}CNMR$ spectra, some transition metals complex 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 prepared complexes were $[M(CAP)_2(H_2O)_2]Cl_2$(M^{+2} =Mn, Co, Ni, Cu, Zn, Cd and Hg),the proposed geometrical structure for all complexes were octahedral.
Key Word :4-Aminoantipyrine, P-ChloroBenzoyl isothiocyanate, complexes.

Introduction

In recent years, there has been increasing interest in synthesis of heterocyclic compounds that have biological and commercial importance. Antipyrine compounds play an important role in modern organic synthesis, not only because they constitute a particularly useful class of heterocyclic compounds, but also because they are of great biological interest. One of the most important derivatives of antipyrine is 4-aminoantipyrine, It is very important in the field of Medicinal and Agricultural chemistry. It is also used as a hemolytic inhibitors, poloro graphic titration, conductometric and potentiometric determination of lanthanides. 4-amino antipyrine have large scale of applications inbiological, clinical, analgesics, antifungal, anti bacterial, anticancereous and pharmacological areas[1-2].Anew metal complexes of Zn (II), Cd(II) and Hg(II) were synthesized from the Schiff base derived from 4-aminoantipyrine, 3-hydroxyl benzaldehyde and ethylene diamine in alcohol medium. [3].A new hexadentate Schiff base

ligand derived from condensation of 4-aminoantipyrine with aromatic diketone and thiosemicarbazide, and its metal complexes Co (II), Ni (II), Cu (II) and Zn (II) were characterized by elemental analyses, molar conductance, magnetic susceptibility measurement and spectral (electronic, IR, UV-Vis, ^{1}HNMR) studies[4].Novel Schiff base and its 3d transition metal complexes of Mn(II), Fe(III) and VO(IV) has been designed and synthesized form 4-aminoantipyrine and Ethyl-4-methyl–oxo-6-phenylhexahyropyrimidine-5-carboxylate[5]. The aim of this work is to prepare and characterize anew ligand [4-chloro-N-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-ylcarbamothioyl)benzamide] (CAP),and it's metal complexes with Mn(II), Co(II), Ni(II), Cu(II), Zn(II), Cd(II) and Hg(II) ions.

Experimental

Chemicals

All reagents were analar or chemical pure grade by BDH, Merck and Fluka. All metal chloride salts and solvents were purchased from Merk and Fluka com., and used without purification.

Instruments

^{1}Hand^{13}C–NMRwere recorded using Ultra Shield 300 MH_z Switzerland and at University of Al al-Bayt, Jordan. 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 spectrophotometer at 25 □C for 10^{-3}M solution DMSO with 1.000 ± 0.001 cm matched quartz cell. Molar Conductivity was measured at 25 □C for 10^{-3}M solution of DMSO by using Philips PW. Digital. micro elemental analysis(C.H.N.S) were performed using Acrlo Erba 1106elemental analyzer. Magnetic susceptibility measurements were obtained by balance magnetic susceptibility model MSB-MKI. Metal contents of the complexes were determined by atomic absorption technique by using Shimadzu (AA680G).

Preparation of ligand(CAP)

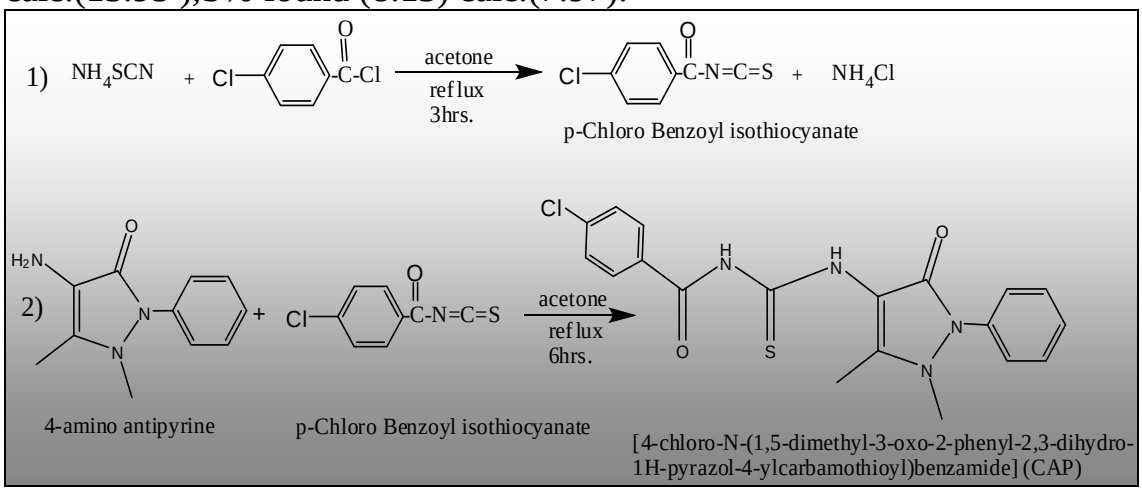
The ligand was prepared by two steps(scheme-1)

(A)- Preparation of the (P-ChloroBenzoyl isothiocyanate)[6]

Mixture of P-ChloroBenzoyl Chloride(3.3 ml,1mmol) and ammonium thiocyanate (2g, 1mmol) in (25 ml) of acetone was stirred under refluxed for 3 hrs and then filtered, the filtrate was used for further reaction.

(B)- Preparation of [4-chloro-N-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-ylcarbamothioyl)benzamide] (CAP)

(5.33g, 1mmol) of 4-aminoantipyrine in (20ml) acetone was rapidly added to P-Chloro Benzoyl isothiocyanate and maintaining reflux. After refluxing for 6 hrs, the resulting solid was collected, washed with acetone and recrystallization from ethanol, yield (80%), (m.p =113-115)˚C**,**C% found (57.1) calc.(56.8), H% found (4.67) calc.(4.48),N% found (13.88) calc.(13.95),S% found (8.13) calc.(7.97).



Synthesis ligand complexes

Synthesis of the $[Cu(CAP)_2(H_2O)_2]Cl_2$ complex

A solution of (0.2g, 1mmol) $CuCl_2.2H_2O$ in (10ml) ethanol was added to solution of (0.803g, 2mmol) (CAP) in (10ml) ethanol. The mixture was stirred for 6 hours at room temperature, the brown solid was collected by filtration, washed with (1:1) mixture of water: ethanol, recrystallized from ethanol and dried in an oven (50˚C).

Synthesis of $[Mn(CAP)_2(H_2O)_2]Cl_2$, $[Co(CAP)_2(H_2O)_2]Cl_2$, $[Ni(CAP)_2(H_2O)_2]Cl_2$, $[Zn(CAP)_2(H_2O)_2]Cl_2$, $[Cd(CAP)_2(H_2O)_2]Cl_2$, $[Hg(CAP)_2(H_2O)_2]Cl_2$ complexes.

A similar method to that mentioned for preparation of $[Cu(CAP)_2(H_2O)_2]Cl_2$ complex was used to prepare the complexes of $[Mn^{+2}, Co^{+2}, Ni^{+2}, Zn^{+2}, Cd^{+2}$ and $Hg^{+2}]$ ions with (CAP),Fig(1) .Table (1) showed some physical properties of the prepared complexes.

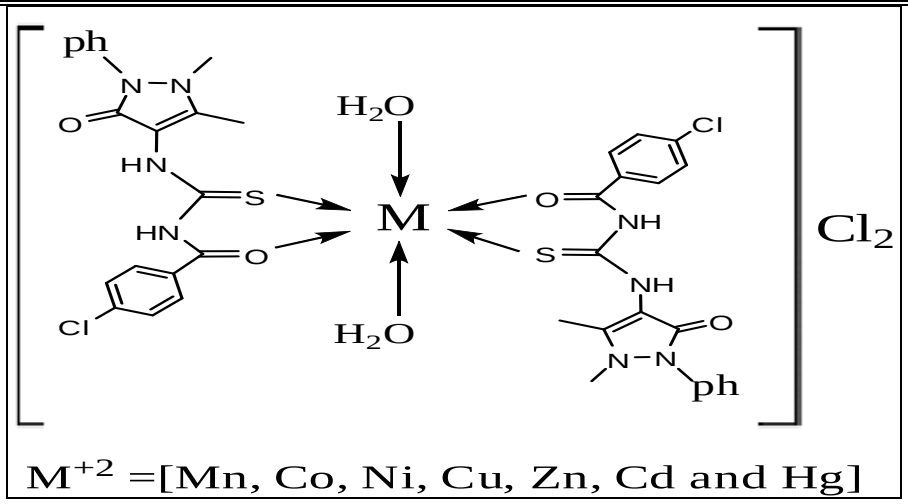


Figure No. (1): The proposed chemical structure formula of the complexes

Resultsand Discussion

Ligand (CAP)

The FT-IR spectrum of the free ligand (CAP) ,Fig.(2) showed bands at(1660) cm^{-1},(1640)cm^{-1} and (1365)cm^{-1} due to υC=O(in ring) , υC=O(amidic) and υC=S respectively. While another absorption band at (3404)cm^{-1} could be explained as υ N-H [7-9].The FT-IR spectral data of the free ligand were listed in Table(2).

The UV-Vis spectrum of the free ligand (CAP). Fig.(4) exhibits a high intense absorption peak at (33783)cm^{-1} which may be attributed to electronic transition type $\pi \longrightarrow \pi^*$[10].The data of electronic spectrum of the free ligand (CAP) were listed in table (3).

The ^{1}H-NMR spectrum of free ligand (CAP),Fig.(6) which was recorded in DMSO-d_6 solvent showed the following signals: singlet at δ(1.2) ppm refers to (3H, CH_3), singlet at δ(2.00) ppm refers to (3H,CH_3N), singlet at δ(3.11) ppm refers to (1H, NH sec. amine), the multiplet signals δ(6.09-7.95) ppm were attributed to aromatic protons, singlet at δ(11.30) ppm refers to (1H,NHsec. amide).

^{13}CNMR spectrum of the free ligand (CAP),Fig.(7)showed chemical shift at δ(12.50) ppm refers (CH_3) for (CH_3CO) group, signal at δ(35.52-35.96) ppm for (CH_3N), signals at δ(37.81-40.38) ppm for DMSO, signal at δ(108.24) ppm refers to (**C**=C-CH_3), The chemical shifts at range δ(122.26-136.02) ppm due to aromatic carbons, signal at δ(137.80) ppm for (=C-N), signal at δ(152.50) ppm for (=C-N, aromatic carbons), while the signals at Fig. (2) showed the following signals at δ(161.25) ppm , δ(167.50) ppm and δ(182.50) ppm were attributed to ((C=O), antipyrine ring), (CONH) and (C=S)[11,12].

Complexes of the ligand(CAP)

The solid complexes soluble in some common solvent such as dimethylformamide, dimethyl sulphoxide and relatively thermally stable. The molar conductivity values of all complexes in DMSO solvent in 10^{-3} M at 25˚C (Table-1) indicated electrolyte nature with 1:2 ratio [13].The atomic absorption measurements for all complexes gave approximated values when its comparison with theoretical values, Table(1) includes the physical properties for the ligand and its complexes.

FT-IR Spectra

These spectra exhibited marked difference between bands Fig. (3) belonging to the stretching vibration of υ(C=O amido) in the range between (1630-1620)cm^{-1}shifted lower frequencies suggesting of 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 (1438-1402)cm^{-1} shifted to higher frequencies which indicates to the coordination of ligand through the sulfur atom at the thiol group to the central ion[15]. The stretching vibration band υ(C=O in ring), υ(N-H) either show no change or very little in their frequencies (1665-1660)cm^{-1},(3380-3178)cm^{-1}respectively there for indicating do not coordinate to the metal ion. Metal-oxygen and metal-sulfur bonds were confirmed by the presence of the stretching tremor of υ(M-O) and υ(M-S) around (501-470)cm^{-1} and(455-420)cm^{-1} respectively the spectra of complexes showed the appearance of bands in the range(879-848)cm^{-1} attributed to υ(OH),these bands confirm the coordination of the water with metal[14],Table (2) describes the important bands and assignment for all prepared complexes.

Magnetic moment

The values of measured magnetic susceptibility and effective magnetic moment (μeff.) for the Mn(II), Co(II), Ni(II), Cu(II)complexes are shown in table(1).Mn(II), Co(II), Ni(II) and Cu(II) complexes exhibit μeff. (5.86, 4.90, 3.06, 1.77) B.M respectively ,which can be a normal values for high spin octahedral complexes.[16]

Electronic spectra for complexes

-$[Mn(CAP)_2(H_2O)_2]Cl_2$ d^5

The Brown complex of Mn(II) shows band at(33898)cm^{-1} due to ligand field and other bands at (27777)cm^{-1} and (13140)cm^{-1} which are caused by the electronic transfer $^6A_1g \longrightarrow {}^4T_2g_{(G)}$ and $^6A_1g \longrightarrow {}^4T_1g_{(G)}$ respectively, suggesting octahedral geometry around Mn(II) ion [17].

-$[Co(CAP)_2(H_2O)_2]Cl_2$ d^7

The spectrum of the Green complex gave four bands at (33898)cm^{-1}, (27397)cm^{-1}, (18083) cm^{-1} and (13157)cm^{-1} attributed to (L.F),C.T mixed with $^4T_1g_{(F)} \longrightarrow {}^4T_1g_{(P)}$, $^4T_1g_{(F)} \longrightarrow {}^4A_2g$ and $^4T_1g_{(F)} \longrightarrow {}^4T_2g_{(F)}$respectively and the rachinter electronic repulsion parameter (B^-) was found to be (400.6)cm^{-1}, from the relation $\beta=B^- / B^0$, was found to be equal (0.79),these parameter are accepted to Co(II) octahedral complex[18].

-$[Ni(CAP)_2(H_2O)_2]Cl_2$ d^8

The spectrum of Brown complex of Ni(II)has revealed the following electronic transfer (L.F),C.T mixed with $^3A_2g \longrightarrow {}^3T_1g_{(P)}$, $^3A_2g \longrightarrow {}^3T_1g_{(F)}$,and$^3A_2g \longrightarrow {}^3T_2g_{(F)}$, transition at (33783)$cm^{-1}$, (28409)$cm^{-1}$, (18518)$cm^{-1}$ and (12642)cm^{-1}respectively, the(B^-) value is found to be (600)cm^{-1},while β was equal to (0.57) these are the characteristics for octahedral complexes of Ni(II)[19].

-[$Cu(CAP)_2(H_2O)_2]Cl_2$ d^9

The spectrum of Brown complex of Cu(II) Fig.(5) shows two bands at (33444)cm^{-1}, (11049) cm^{-1} caused to (C.T), $^2Eg \longrightarrow {}^2T_2g$ transition respectively ,which was a good agreement for distorted octahedral complex for Cu(II) ion[20,21].

-$[Zn(CAP)_2(H_2O)_2]Cl_2$,$[Cd(CAP)_2(H_2O)_2]Cl_2$ and $[Hg\ (CAP)_2\ (H_2O)_2\ ]Cl_2$

Show only charge transfer of (M→L) in range (22898-33783)cm^{-1}[22,23]. All transition 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}C-NMR for ligand (CAP)only)for the ligand and all prepared complexes, we suggested that the ligand (CAP) behaves as bidentate 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 sulfer atom of (C=S) group, suggesting octahedral geometry around metal ions for all prepared complexes.

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

Compounds	M.wt (gm/mole)	Color	M.P(□C) or dec.	M% Calculation (Found)	Molar Cond. $Ohm^{-1}cm^2mol^{-1}$ in DMSO	μ_{eff} (B.M)
$C_{19}H_{18}N_4O_2SCl$ CAP	401.5	Brown	113-115 □C	–	5.32	–
$[Mn(CAP)_2(H_2O)_2]Cl_2$	963.94	Brown	206-208 □C	5.70 (5.76)	72.45	5.86
$[Co(CAP)_2(H_2O)_2]Cl_2$	967.93	Green	186-188 □C	6.08 (6.19)	70.12	4.90
$[Ni(CAP)_2(H_2O)_2]Cl_2$	967.71	Brown	220 dec.	6.06 (6.34)	86.32	3.06
$[Cu(CAP)_2(H_2O)_2]Cl_2$	972.54	Brown	125-127 □C	6.53 (7.12)	75.60	1.77
$[Zn(CAP)_2(H_2O)_2]Cl_2$	974.38	Brown	137-139 □C	6.71 (7.01)	71.2	0
$[Cd(CAP)_2(H_2O)_2]Cl_2$	1021.41	Brown	122-124 □C	11.00 (10.65)	74.6	0
$[Hg(CAP)_2(H_2O)_2]Cl_2$	1109.59	Brown	230 dec.	18.08 (17.93)	84	0

dec.=decomp

Table No. (2): The characteristic infrared band for free ligand (CAP) and its metal complexes

Compounds	υ(N-H)	υ(C=O) in Ring	υ(C=O) Amide	υ(C=S)	υ(O-H)	υ(M-O)	υ(M-S)
Ligand CAP	3404 (w)	1660 (s)	1640(w)	1365(S)	–	–	–
$[Mn(CAP)_2(H_2O)_2]Cl_2$	3332(w)	1660(m)	1625(s)	1404(w)	855(s)	470(m)	455(w)
$[Co(CAP)_2(H_2O)_2]Cl_2$	3332(w)	1660(s)	1630(w)	1430(w)	848(s)	470(w)	425(m)
$[Ni(CAP)_2(H_2O)_2]Cl_2$	3380(w)	1660(m)	1620(s)	1418(m)	848(m)	473(m)	432(w)
$[Cu(CAP)_2(H_2O)_2]Cl_2$	3282(w)	1660(s)	1630(w)	1402(s)	879(m)	475(m)	432(w)
$[Zn(CAP)_2(H_2O)_2]Cl_2$	3317(m)	1660(s)	1623(w)	1438(m)	850(s)	470(m)	425(m)
$[Cd(CAP)_2(H_2O)_2]Cl_2$	3375(m)	1660(s)	1625(m)	1414(m)	848(m)	475(m)	420 (m)
$[Hg(CAP)_2(H_2O)_2]Cl_2$	3178(m)	1665(s)	1630(w)	1419(w)	848(s)	501(m)	455(m)

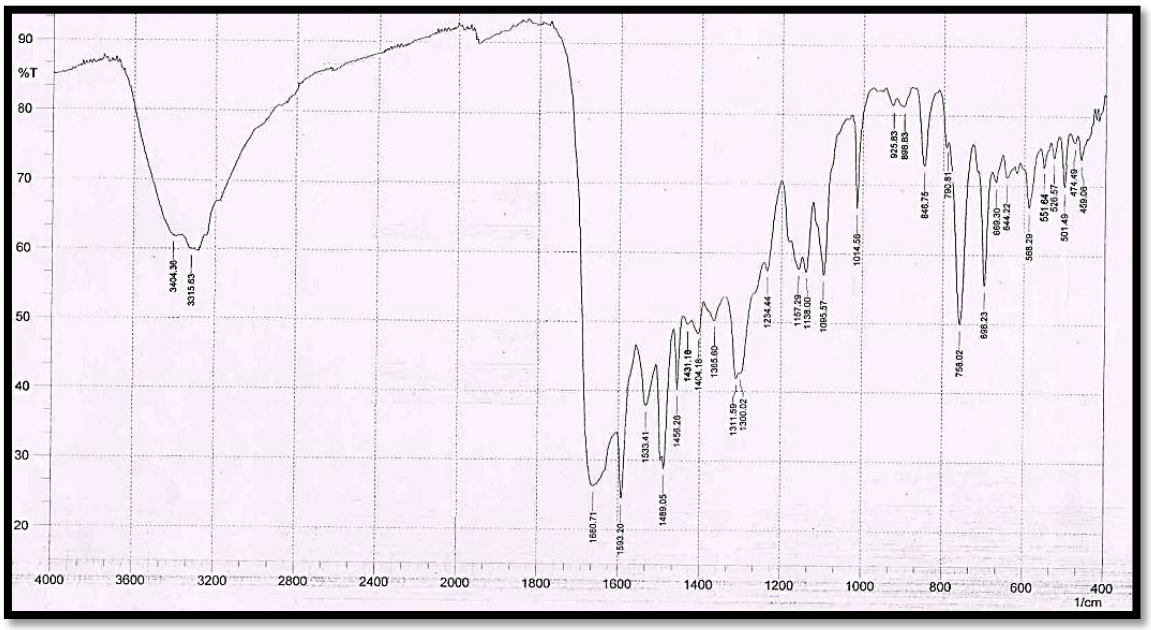

Figure No. (2): Infrared spectrum of ligand (CAP)

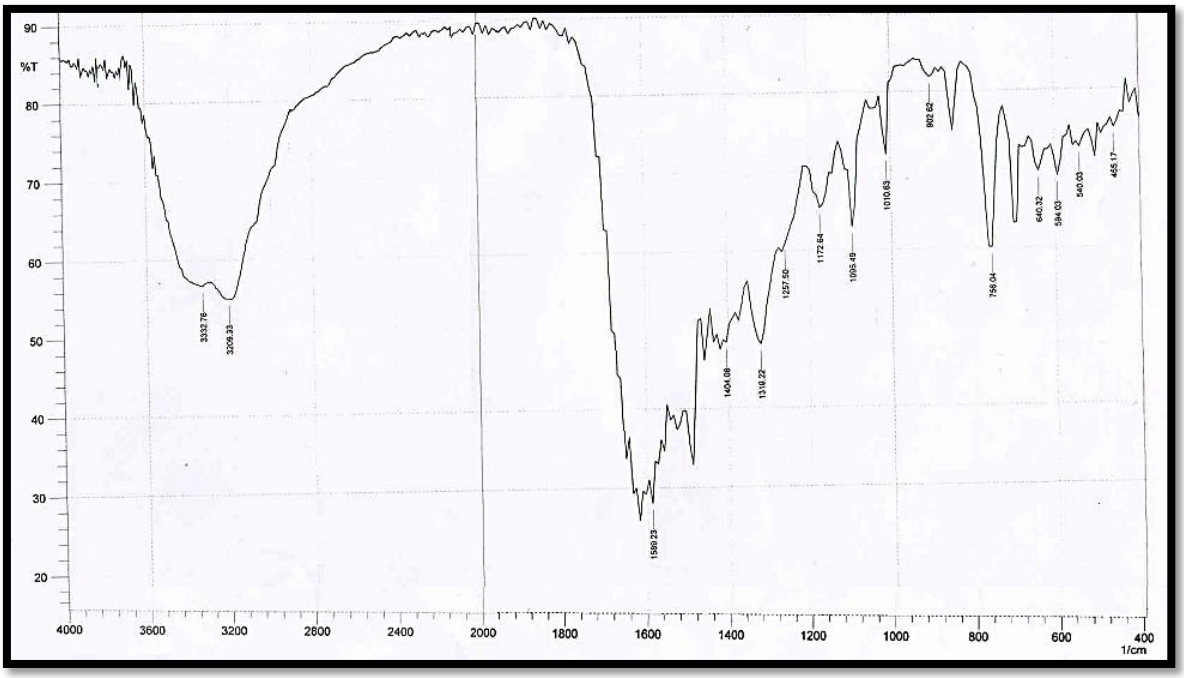

Figure No. (3):Infrared spectrum of complex [Mn $(CAP)_2(H_2O)_2]Cl_2$

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

Compounds	λ_{max} nm	Wave number cm^{-1}	ABS	ε_{max} $molar^{-1}cm^{-1}$	Transitions
Ligand CAP	296	33783	2.069	2069	$\pi \longrightarrow \pi^*$
$[Mn(CAP)_2(H_2O)_2]Cl_2$	295 360 761	33898 27777 13140	2.001 0.500 0.010	2001 500 10	L.F $^6A_1g \longrightarrow {}^4T_2g_{(G)}$ $^6A_1g \longrightarrow {}^4T_1g_{(G)}$
$[Co(CAP)_2(H_2O)_2]Cl_2$	295 365 553 760	33898 27397 18083 13157	2.040 0.500 0.075 0.010	2040 500 75 10	L.F C.T mixed with $^4T_1g_{(F)} \longrightarrow {}^4T_1g_{(P)}$ $^4T_1g_{(F)} \longrightarrow {}^4A_2g$ $^4T_1g_{(F)} \longrightarrow {}^4T_2g_{(F)}$
$[Ni(CAP)_2(H_2O)_2]Cl_2$	296 352 540 791	33783 28409 18518 12642	2.151 0.728 0.100 0.020	2151 728 100 20	L.F C.T mixed with $^3A_2g \longrightarrow {}^3T_1g_{(P)}$ $^3A_2g \longrightarrow {}^3T_1g_{(F)}$ $^3A_2g \longrightarrow {}^3T_2g_{(F)}$
$[Cu(CAP)_2(H_2O)_2]Cl_2$	299 905	33444 11049	2.418 0.036	2418 36	C.T $^2Eg \longrightarrow {}^2T_2g$
$[Zn(CAP)_2(H_2O)_2]Cl_2$	295	33898	2.085	2085	C.T
$[Cd(CAP)_2(H_2O)_2]Cl_2$	296	33783	2.172	2172	C.T
$[Hg(CAP)_2(H_2O)_2]Cl_2$	295	33898	2.106	2106	C.T

C.T = Charge transfer

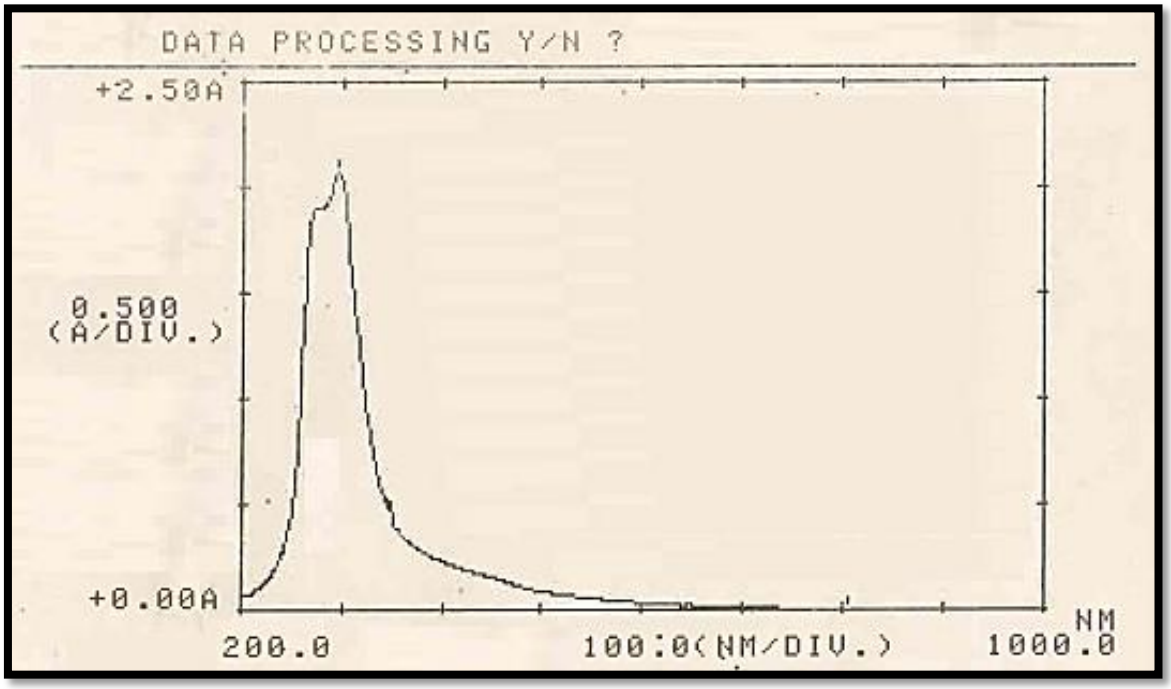

Figure No. (4):U.V. spectrum of ligand (CAP)

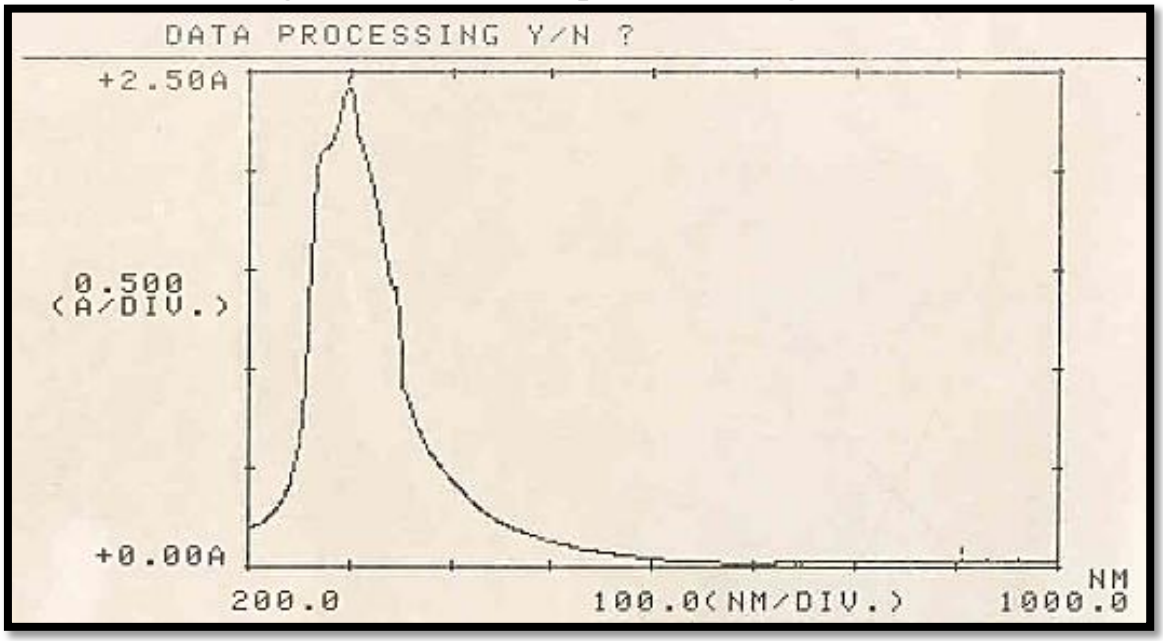

Figure No. (5): U.V. spectrum of complex $[Cu(CAP)_2(H_2O)_2]Cl_2$

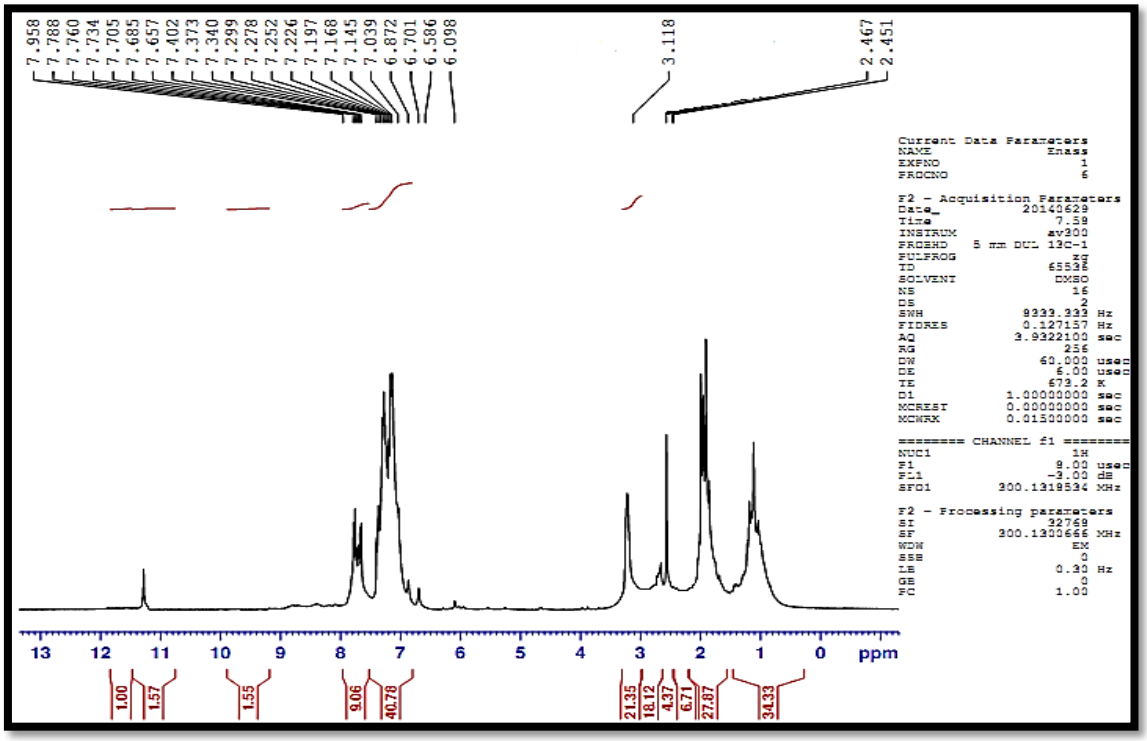

Figure No. (6):^{1}H-NMRspectrum of ligand (CAP)

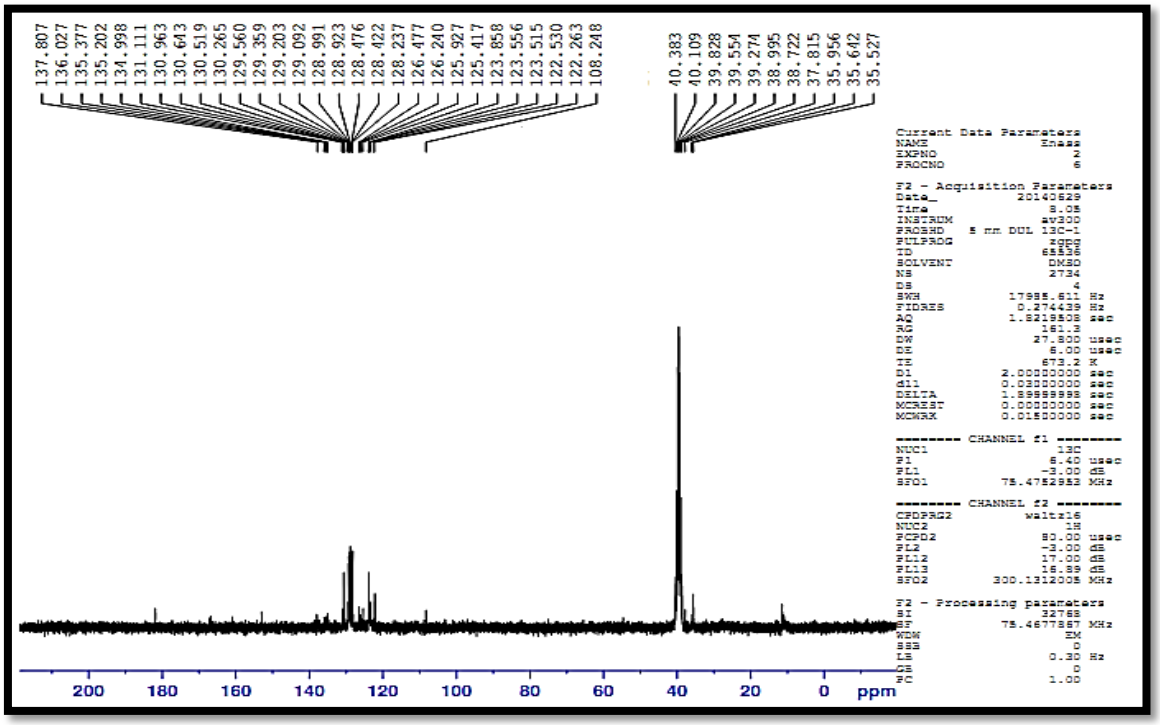

Figure No. (7):^{13}C-NMR spectrum of ligand (CAP)

تحضير وتشخيص بعض المعقدات الفلزية مع -(5,1- داي مثيل-3-اوكسو-2-فنيل-3,2-داي هايدرو-1- بايرازول-4-يل كاربامو ثايويل)بنزمايد4N-كلورو-

باسمة محسن سرحان إيناس جاسم وحيد

قسم الكيمياء/ كلية التربية للعلوم الصرفة (ابن الهيثم) / جامعة بغداد

عوف عبد الرحمن احمد

وزارة التربية- العراق

الخلاصة

حضر الليكاند الجديد **-(5,1- داي مثيل-3-اوكسو-2-فنيل-3,2-داي هايدرو-1-بايرازول-4-يل كاربامو ثايويل)بنزمايد4N-كلورو-**

وذلك من مفاعله (بارا- كلوروبنزويل ايزوثايوسيانات) مع 4-امينو أنتي بايرين وبنسبة (1:1) وشخص بوساطة التحليل الدقيق للعناصر(C.H.N.S.) والأشعة تحت الحمراء والأشعة فوق البنفسجية- المرئية وطيف الرنين النووي المغناطيسي ، كما حضرت وشخصت معقدات أملاح بعض ايونات العناصر الانتقالية الثنائية التكافؤ (Hg, Cd, Zn, Cu, Ni, Co, Mn) مع الليكاند (CAP) وشخصت المعقدات المحضرة باستعمال الأشعة تحت الحمراء والأشعة فوق البنفسجية – المرئية والتوصيلية المولارية والحساسية المغناطيسية والامتصاص الذري واستنتج من الدراسات والتشخيصات إن المعقدات لها شكل ثماني السطوح حول الايون الفلزي مع اللكياند (CAP) ثنائي السن.

الكلمات المفتاحية: 4-امينو انتيبايرين، بارا- كلوروبنزويل ايزوثايوسيانات، معقدات.