*Synthesis and Characterization of Some New Metals Complexes of [N-(4-chlorobenzoyl amino)-thioxomethyl] Anthranilic Acid (CBA)

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Abstract

A new ligand [N-(4-chloro benzoyl amino)-thioxo methyl] anthranilic acid (CBA) was synthesized by reaction of 4chlorobenzoyl isothiocyanate with anthranilic acid by using acetone as a solvent , The ligand was characterized by elemental analysis ,FT-IR,and 13 C, 1 H-NMR spectra, some transition metals complexes of this ligand was prepared and characterized by FT-IR, UV-Visible spectra , conductivity measurements , magnetic susceptibility , atomic absorption and molar ratio (M:L). From results obtained ,the following formula [M (CBA $)_2$] where $M^{+2}=Mn$, Co , Ni , Cu , Pd , Zn , Cd ,and Hg , the proposed molecular structure for these complexes as tetrahedral geometry except copper and palladium complexes are have square planer geometry .

Key words: Anthranilic acid, 4-chloro benzoyl isothiocyanate, complexes.

Introduction

Anthranilic acid is a constituent of many of the bioactive compounds that exhibits a rang of biological activities specifically, the nucleus of anthranilic acid biochemical precursor to the amino acid and its derivatives as well as constituents of several a lkaloids⁽¹⁾ and also anthranilic acid and their derivatives with metal complexes have great importance

In biological fields such as antitumor $^{(2)}$, antifungal, antibacterial $^{(3)}$, and inhibition effect for corrosion of copper and copper alloys in different media $^{(4)}$

A new series of dyes were synthesized from anthranilic acid derivatives⁽⁵⁾. Irudya.A.sophia .and coworkers⁽⁶⁾were reported the synthesis and characterization of poly anthranilic acid

Metal Nanocomposites and the complex formation of Am(III) with anthranilic acid was studied at pH 3.8 and 6.0 at an ionic strength of 0.1M $(NaClO_4)^{(7)}$. also new monocyclopentadienyl titanium complexes with functionalized carboxylate ligands as anthranilic acid have been synthesized⁽⁸⁾. The aim of this work preparation some new transition metal complexes of [N-(4-chlorobenzoyl amino)-thioxomethyl] anthranilic acid (CBA).

*The Research is apart of on Ph.D. dissertation in the case of the second researcher

Experimental

Chemicals: All reagents were analar or chemical puregrade by BDH and Fluka . **Materials:** (4- Methoxy benzoyl chloride) ,(Anthranilic acid) (Fluka), Manganese chloride tetrahydrate (MnCl₂ \cdot 4H₂O), Cobalt chloride hexahydrat (CoCl₂ \cdot 6H₂O), Nickel chloride hexahydrat (NiCl₂ \cdot 6H₂O), Copper chloride dehydrate (CuCl₂ \cdot 2H₂O), Zinc chloride (ZnCl₂), Cadmium chloride hydrate (CdCl₂ \cdot H₂O) and Mercury chloride (HgCl₂).

Instruments:

 $^1\mathrm{H}$ and $^{13}\mathrm{C}$ –NMR was recorded using Ultra Shield 300 MHz Switzerland at university of Al al-Bayt , Jordan by using DMSO-d6 . Melting point was recorded by using stuart- melting point apparatus . FT-lR spectra were recorded as KBr disc using Shimadzu in the range of 400-4000 cm $^{-1}$. Electronic spectra were obtained using UV Shimadzu spectra photometer and Conductivity was measured by using Philips Pw. Digital. at 25 C 0 in $10^{-3}\mathrm{M}$ DMSO

Elemental analyses (C.H.N.S) were performed using acrloErba 1106 elemental analyzer. Magnetic susceptibility measurements were obtained by Balance magnetic susceptibility by model MSB-MKl. Metal contents of the complexes were determined by atomic absorption technique by using shimadzu (AA680G).

Preparation of the ligand (CBA)

1-Preparation of the (4-cloro benzoyl isothiocyanate):

Mixture of 4-chloro benzoyl chloride(3.33 ml),(1mmol) and ammonium thiocyanate (2g) , (1mmol) in (25 ml) of acetone was stirred under refluxed for 3 hours and then filtered ,the filtrate was Yield (85%) , (m.p =188-190) C^0 used for furthera reaction (9).

2- preparation of $[N-(chlorobenzoyl\ amino)thioxomethyl]$ anthranilic acid(CBA):

(3.60~g)~(1mmol~) of anthranilic 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 (75%) , (m.p =218-220) C^0 , %C found (53.37) while calculate(53.81) , %H found (3.04) while calculate (3.28) ,%N Found (8.67) while calculate (8.37) ,%S found (8.21) while calculate(9.56).

Synthesis of metal complexes

(0.67~g , 2mmole) of ligand (CBA) was dissolved in 25ml of ethanol containing (0.12g , 2mmole) of KOH, then the solution of following metal salts MnCl $_2$.4H $_2$ O (0.2g , 1mmole), CoCl $_2$.6H $_2$ O (0.24g ,1mmole),NiCl $_2$.6H $_2$ O (0.24g,1mmole) ,CuCl $_2$.2H $_2$ O (0.2g ,1mmole), ZnCl $_2$ (0.14g, 1mmole),CdCl $_2$.H $_2$ O (0.2g ,1mmole), HgCl $_2$ (0.3g ,1mmole) and PdCl $_2$ (0.2g , 1mmole) in ethanol , were added dropwise to the solution of the ligand (CBA-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) .

Scheme(1): preparation of (CBA)

Results and Discussion

The solid complexes, 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 the ligand and it's complexes.

Spectral studies:

$^{1\bar{3}}C$, ^{1}H NMR spectra :

1-¹H NMR spectrum for the ligand (CBA) in DMSO-d6 as solvent Figure (1) showed the following signals:

singlet at $\delta(2.08-2.73)$ ppm for DMSO-d6, broad at $\delta(3.07-3.75)$ ppm for (1H, NH sec,amine), pairs of doublet at $\delta(7.35-7.73)$ ppm for (4H,aromatic protons), multiplet at $\delta(7.93-8.19)$ ppm for (4H, aromatic protons), singlet at $\delta(11.65)$ ppm for (1H,NH sec,amide), singlet at $\delta(13.12)$ ppm for (1H,COOH).

2-the ¹³C NMR spectrum of (CBA) in DMSO figure (2) showed the Following signals:

signals at δ (38.69-40.80) ppm for DMSO, signals at δ (124.64-138.10) ppm for aromatic carbons, signal at δ (166.53) ppm for (C=O sec. amide), signal at δ (167.07) ppm for (COOH), signal at δ (179.59) ppm for C=S).

Infrared spectra:

FT-IR spectrum of the free ligand (CBA) figure (3) , Showed bands due to amido v(NH + OH) ,v(C=O) and v(C=S) which absorbed at (3031 +3471)cm⁻¹ , (1681)Cm⁻¹ , and (1257)Cm⁻¹ respectively , while another absorption band appeared at (1698)cm⁻¹ could be explained as v(COO)_{asym} (11, 12), where the v(OCO)_{sym} was noticed at (1411)cm⁻¹.

The FT-IR spectra of complexes:

These spectra figure(4 - 12) exhibited marked difference between bands belonging to the stretching vibration of v(NH) of the amine group in the range between (3425-3301)cm⁻¹ shifted higher frequencies by (394-270)cm⁻¹ suggesting of the possibility of the coordination of ligand through the nitrogen atom at the amine group (13). absorption assigned for v(COO)_{asym} was noticed at the range (1582-1527)cm⁻¹ shifted to higher frequencies by (180-171)cm⁻¹ while the band caused by v(COO)_{sym} appeared between (1518-1450)cm⁻¹ by (107-39)cm⁻¹ which to lower frequencies indicates to the coordination of the carboxylic group to the central ion⁽¹⁴⁾. the stretching vibration band v(C=O) and v(C=S) carbonyl group either show no change or very little in their frequencies (1681-1635)cm⁻¹ and (1288-1249)cm⁻¹ respectively there for indicating do not coordinate to the metal ion⁽¹⁵⁾ . Metal-nitrogen and metal-oxygen bonds were confirmed by the presence of the stretching vibration of v(M-O) and v(M-N) around (416-478)Cm⁻¹ and (439-532)Cm⁻¹ respectively ,Table (2) describe the important bands and assignment for free ligand (CBA) and it's complexes.

Electronic spectral:

The UV-Visible of the ligand (CBA) and its complexes recorded in Table (3) the solution of the ligand (CBA)in 10^{-3} M (DMSO) exhibited two peaks figure(5) at (321)nm and (268)nm which are attributed to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ Transition respectively⁽¹⁶⁾.

The spectra of complexes:

- -[Mn(CBA)₂] d^5 : the pale yellow complex of Mn (TI) shows bands at (36363)cm⁻¹ and (30581)cm⁻¹ due to charge transfer and another bands at (11261)cm⁻¹ and (10121)cm⁻¹ which are caused by the electronic transfer $^6A_1 \rightarrow ^4T_2(D)$ and $^6A_1 \rightarrow ^4T_1(D)$ respectively $^{(17)}$.
- -[Co(CBA)2] \textbf{d}^7 : the spectrum of the green complex figure(7) gave four bands at (37878)cm $^{-1}$, (24691)cm $^{-1}$, (16129)cm $^{-1}$ and (11261)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 , and the rach interelectronic repulsion parameter (B $^-$) was found to be (469)cm $^{-1}$, from the relation $\beta = B^- \ / \ B_0$, was found to be a equal (0.48) ,these parameter are accepted to Co(TI) tetrahedral complex $^{(18)}$.
- -[Ni(CBA)2] d^8 : the spectrum of deep green complex of Ni(TI) has revealed the following electronic transfer (C.T.),
- $^3T_1(F) \!\!\to^3\!\! T_1(P)$, $^3T_1(F) \!\!\to^3\!\! A_2(F)$,and $^3T_1 \!\!\to^3\!\! T_2(F)$, transition at (37735)cm⁻¹ , (25974)cm⁻¹ , (15748)cm⁻¹ and (11587)cm⁻¹Respectively , the(B^-) value found to be (464)cm⁻¹ ,while β was equal to (0.445) these are the characteristics for tetrahedral complexes of Ni (TI) $^{(-19)}$.

- -[$Cu~(CBA)_2$] d^9 : the spectrum of brawn complex of Cu(TI) show three bands at $(37878)cm^{-1}$, $(17667)cm^{-1}$ and $(17452)cm^{-1}$ caused to (C.T), $^2B_{1g}{\to}^2A_{1g}$ and $^2B_{1g}{\to}^2B_{2g}$ transition respectively $^{(20)}$.
- -[Pd (CBA)2] d^8 : the spectrum of brawn complex of Pd(TI) gave three bands at (37313)cm⁻¹, (11286)cm⁻¹ and (10142)cm⁻¹ attributed to (C.T), $^1A_{1g} \rightarrow ^1A_{2g}$, $^1A_{1g} \rightarrow ^1B_{1g}$ transition respectively (18) .
- the complexes of [Zn(CBA)₂] , [Cd(CBA)₂] and [Hg(CBA)₂] shows only charge transfer of (M \rightarrow L) in range (37453-30864)cm^{-1 (21)} . All transition with their assignments are summarized in table (3) .

Study of Complexes Formation in Solution:

Complexes of ligand (CBA) with metal ions were studied in solution using ethanol as solvent in order to determine [M/L] ratio in complexes follow molar Method $^{(22)}$. A series of solution were prepared having A constant concentration (10 $^{-3}$ M) of metal ion and ligand. The [M/L] ratio 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(CBA) $_2$] where $M^{+2} \!\!=\!\! (Mn^{+2}$, Co^{+2} , Ni^{+2} , Zn^{+2} , Cd^{+2} and Hg^{+2}) while copper and palladium complexes have square planer .

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Table (1): Some physical properties of the ligand (CBA) and its complexes

Compound	M.Wt (gm/mole)	Color	M.P(C°)	M % Calculation (found)	Molar Condu. Ohm¹cm²mol¹- in DMSO	µeff(B.M)
(CBA)	334.77	Pale Yellow	220-224		2.5	
Mn (CBA) ₂	722.48	Pale Yellow	190	7.60 (6.95)	13.4	5.67
Co(CBA) ₂	726.47	Green	215-216	8.11 (7.77)	10.3	4.82
Ni(CBA) ₂	726.23	Deep Green	240(dec)	8.08 (7.65)	7.9	2.94
Cu(CBA) ₂	731.08	Dark brawn	232	8.69 (8.10)	20.1	1.72
Zn(CBA) ₂	732.92	Pale Yellow	190-192	8.92 (8.32)	8.5	
Cd(CBA) ₂	779.95	Pale Yellow	250	14.41 (15.10)	16.8	
pd(CBA) ₂	773.96	brawn	270(dec)	13.76 (13.12)	12.1	
Hg(CBA) ₂	868.13	Dark gry	185 (dec)	23.10 (22.85)	5.2	

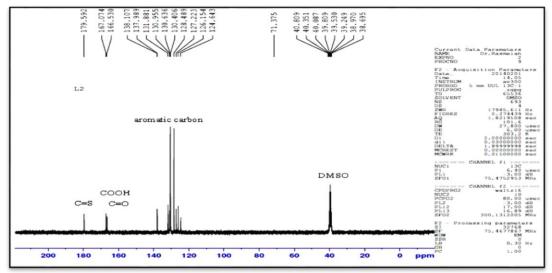
Table (2):- the characteristic infrared band for free ligand (CBA) and its metal complexes

Compound	υ(N-H) υ(O—H)	υ(COO) _{smm}	υ(COO) _{a smm}	υ(C=O)	υ (C=S)	υ(M-O)	υ (M—N)
CBA=Ligand	3031 (b) 3471 (s)	1411 (s)	1698(w)	1681 (s)	1257(s)	_	_
[Mn(CBA) ₂]	3417 (b)	1504 (s)	1589 (s)	1681 (m)	1249 (s)	470 (m)	439 (w)
[Co(CBA)2]	3394 (m)	1458(s)	1527 (s)	1650 (w)	1296 (s)	478 (w)	416 (m)
[Ni(CBA)2]	3301(b)	1450(m)	1589 (s)	1635 (m)	1296 (s)	478 (m)	455 (w)
[Cu(CBA)2]	3409(b)	1512(s)	1589 (s)	1681 (w)	1272 (m)	439 (m)	416 (m)
[Zn(CBA) ₂]	3394 (b)	1519(s)	1589 (s)	1681 (m)	1257 (s)	478 (m)	455 (w)
[Cd(CBA)2]	3425 (b)	1512 (s)	1589 (s)	1681 (s)	1288 (s)	524(w)	457(w)
[Hg(CBA)2]	3286 (s)	1473(m)	1527 (s)	1650 (s)	1288 (m)	532 (m)	478 (m)
[Pd(CBA) ₂]	3417 (b)	1504 (s)	1589 (s)	1681 (w)	1249 (w)	516 (w)	478 (m)

Table (3): The peaks, electronic transitions and structure geometries of the ligand (CBA) and its complexes

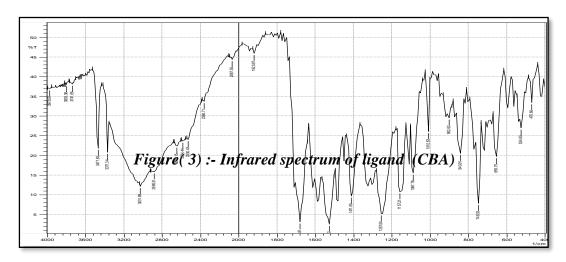
compounds	$\lambda(nm)$	v ⁻ (cm ⁻ 1)	ABC	€ max	Transitions
ligand L ₂ (CBA)	268 321	37313 31152	1.440 0.629	1440 629	π-π* n-π*
Mn(CBA) ₂	275 327 888 988	36363 30581 11261 10121	1.340 0.690 0.010 0.050	1340 690 10 50	$C.T$ $C.T$ ${}^{6}A_{1} \rightarrow {}^{4}T_{2}(D)$ ${}^{6}A_{1} \rightarrow {}^{4}T_{2}(D)$
Co(CBA) ₂	264 405 620 888	37878 24691 16129 11261	2.301 0.035 0.020 0.015	2301 35 20 15	C.T ${}^{4}A_{2}(F) \rightarrow {}^{4}T_{1}(P) {}^{4}A_{2}(F) \rightarrow {}^{4}T_{1}(F)$ ${}^{4}A_{2}(F) \rightarrow {}^{4}T_{2}(F)$
Ni(CBA) ₂	265 385 635 863	37735 25974 15748 11587	1.590 0.490 0.015 0.010	1590 490 15 10	C.T $^{3}T_{1}(F) \rightarrow ^{3}T_{1}(P)$ $^{3}T_{1}(F) \rightarrow ^{3}A_{2}(F)$ $^{3}T_{1}(F) \rightarrow ^{3}T_{2}(F)$
Cu(CBA) ₂	264 566 573	37878 17667 17452	2.000 0.01 0.009	2000 10 9	$C.T$ ${}^{2}B_{1}g \rightarrow {}^{2}A_{1}g$ ${}^{2}B_{1}g \rightarrow {}^{2}B_{2}g$
Zn(CBA) ₂	268	37174	1.545	1545	С.Т
Cd(CBA) ₂	267 324	37453 30864	1.579 0.963	1573 963	C.T C.T
Pd(CBA) ₂	268 886 986	37313 11286 10142	1.400 0.050 0.070	1400 50 70	$\begin{array}{c} \text{C.T} \\ {}^{1}\text{A}_{1}\text{g} \rightarrow {}^{1}\text{A}_{2}\text{g} \\ {}^{1}\text{A}_{1}\text{g} \rightarrow {}^{1}\text{B}_{1}\text{g} \end{array}$
Hg(CBA) ₂	267	37453	1.214	1214	С.Т

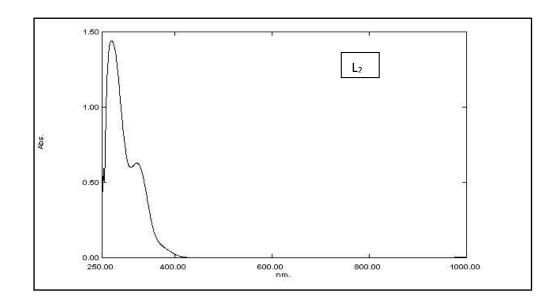
C.T = Charge transfer



Processing parameters | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.000 | 1.0

Figure(2):- ¹H-NMR spectrum of ligand (CBA).





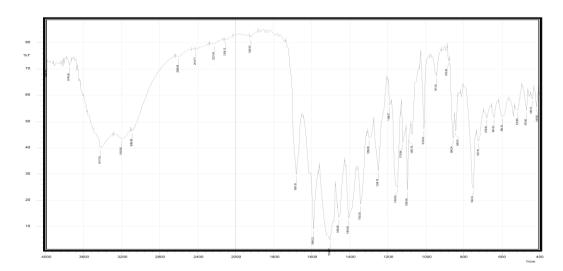


Figure (5):- Infrared spectrum of ligand $Mn(CBA)_2$.

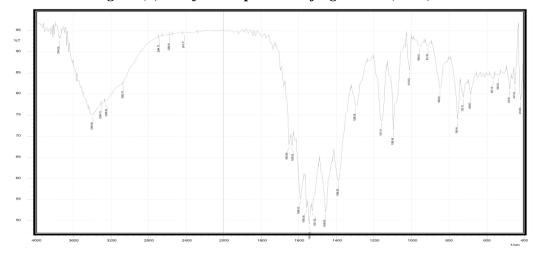


Figure (6): Infrared spectrum of ligand Co(CBA)₂.

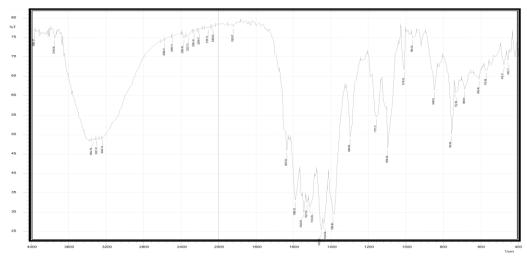


Figure (7): Infrared spectrum of ligand $Ni(CBA)_2$.

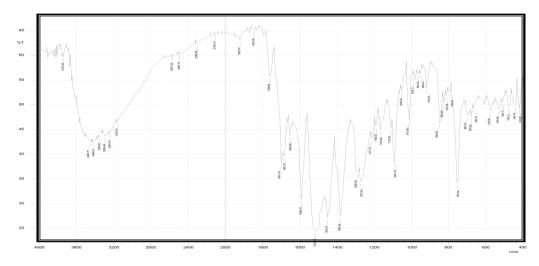


Figure (8):- Infrared spectrum of Cu(CBA)₂.

82.5

87.7

87.5

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Figure (9): Infrared spectrum of ligand $Zn(CBA)_2$.

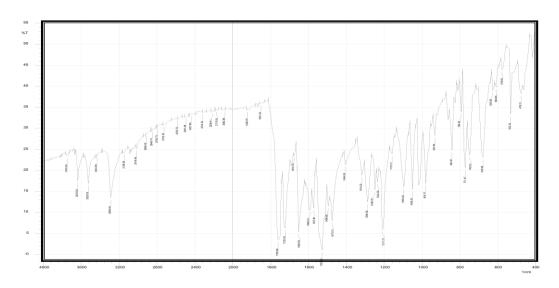


Figure (10): Infrared spectrum of ligand Hg(CBA)₂.

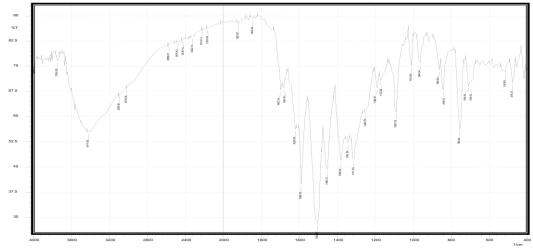


Figure (11):- Infrared spectrum of ligand Pd(CBA)₂

Figure (12):- U.V spectrum of Mn(CBA)₂ complex

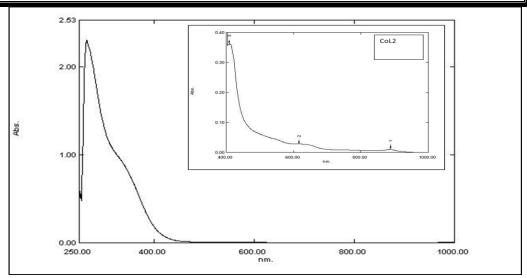


Figure (13):- U.V spectrum of Co(CBA)₂ complex

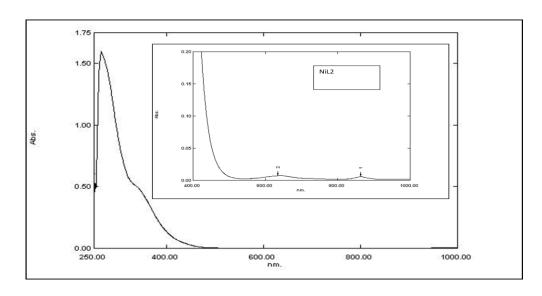


Figure (14):- U.V spectrum of Ni(CBA)₂ complex

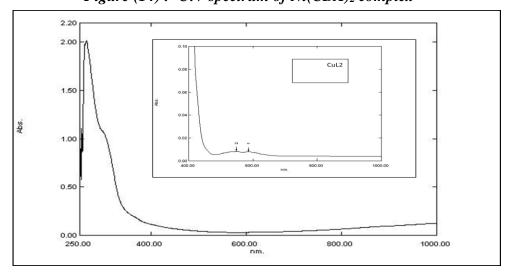


Figure (15) :- U.V spectrum of Cu(CBA)₂ complex

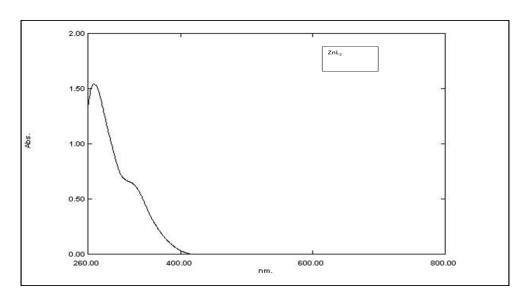


Figure (16): - U.V spectrum of Zn(CBA)₂ complex

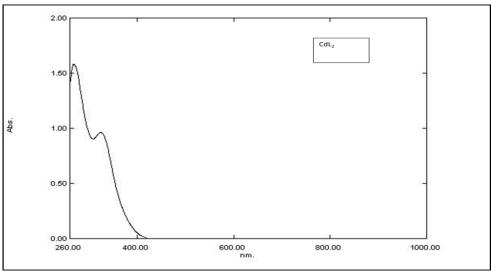


Figure (17): - U.V spectrum of Cd(CBA)₂ complex

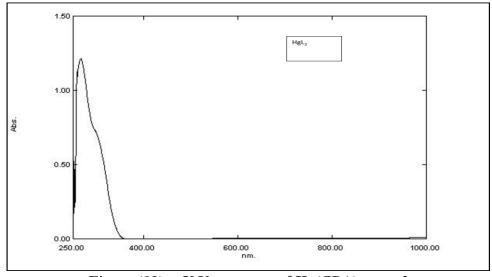
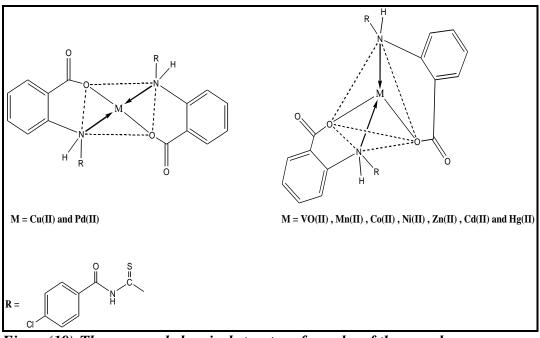


Figure (18): - U.V spectrum of Hg(CBA)₂ complex



Figure(19): The proposed chemical structure formula of the complexes

تحضير وتشخيص بعض المعقدات الفلزية الجديدة مع N-(4] كلورو بنزويل امينو - ثايواوكسو مثيل M-(4) الانثرانلك

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لخلاصة

حضر الليكاند الجديد (4)-N](CBA) كلورو بنزويل امينو-ثايواوكسو مثيل)] الانثرانلك و ذلك من مفاعلة (4-كلورو بنزويل ايزوثايوسيانات) مع حامض الانثراللك وبنسبة (1:1) وشخص بوساطة التحليل الدقيق للعناصر (S.N.H.C) والأشعة تحت الحمراء والأشعة فوق البنفسجية المرئية وطيف الرنين النووي المغناطيسي، كما حضرت وشخصت معقدات بعض ايونات العناصر الانتقالية الثنائية التكافؤ (CBA, Zn) مع الليكاند (CBA) 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 اللكياند (CBA) ثنائي السن ماعدا معقدي النحاس والبلاديوم الذين أعطي الشكل المربع المستوي .

الكلمات المفتاحية: حامض الانثرانلك ، 4-كلوروبنزويل ايزوثايوسيانيت ،معقدات.

البحث مستل من اطروحة دكتوراه للباحث الثاني.