Synthesis and Spectroscopic Studies of Some Metal Complexes of [3-(3-(2-chloroacetyl)thioureido)pyrazine-2-carboxylic acid]

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Abstract

A new ligand [3(3(2chloroacetyl) thioureido)pyrazine-2-carboxyli cacid](CPC)was synthesized by reaction of rized by micro elmental analysis C.H.N.S.,FT-IR,UV-Vis and ^1H-^{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[$M(CPC)_2$](M^{+2} =Mn. Co, Ni, Cu, Zn, Cd and Hg),the proposed geometrical structure for all complexes were as tetrahedral geometry except copper complex has square planer geometry.

Key Word: 3-Amino pyrazine-2-carboxelic acid, chloroacetyl isothiocyanate, complexes

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Introduction

Heterocyclic compounds are abundant in nature andare of great significance to life Because their is in many natural products such, hormones, antibiotics....etc. Apractical method for the synthesis of such compounds is of great interestn synthetic organic chemistry [1].Pyrazine derivativesoccur in many natural sources and can be synthesized chemically or biologically.In animals and plants[2].pyrazine and carboxylate groups are akind of interesting ligands for the coordination of silver, and such complexes have received particular attention in coordination chemistry[3]. It is know that pyrazines and their derivatives from quite important class of compounds present in many natueal flavours and compound organic molecules [4] Also,few examples of pyrazine -carboxylic acid and is derivatives are Known as good building blocks for construction of novel meta orgnic f rame works thatfacilitate the formation of supra molecular architectures [5,6]. An additional amino substitution on 3-amino pyrazine-2-carboxylic acid could be expected to enhance crystal packing through extensive hydrogen bonding The dynamic pattern of 3-amino pyrazinr-2-carboxylic acid by inelastic and incoherent neutron scattering,Raman spectroscopy and abignition.calculation have been reported [7]. X-ray crystal structure of Mg (II)andCa(II)3-aminopyrazinr-2-carboxylic acid complexes havebeen reported [8].Inthisstudy the chemistry of 3-aminopyrazin-2-carboxylic acid .the disociable carboxylic proton with some transition metal ions are reported .These complexes Have been characterization on bases of the spectral(IR,^{1}HNMR,electronic and (ESR)conductivityand magnetic measuremen-ts. New complexes was prepared by the reactionof3-aminopyrazin-2-carboxelic acid acide,withVO^{2+},Pd(II),W(VI) and UO_2^{2+} [9].The aim of this work is to prepare and characterize a new ligand[3-(3-(2-chloro acetyl) thioureido)pyrazine-2-carboxylicacid] (CPC),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 chemicals were supplied from Al-Drich, Fluka and BDH.

Materials: (chloro acetyl chloride,3-amino pyrazin-2-carboxelicacid) tetra hydrate ($MnCl_2.4H_2O$) , Cobalt chloride hexahydrate ($CoCl_2.6H_2O$),Nickel chloride hydrate ($NiCl_2.6H_2O$) 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 were recorded using Ultra 300 MH_z Switzerland at University of Al al-Bayt,Jordan.Melting point wasrecorded by using Stuart- melting point apparatusFTIRspectra were recorded as KBr discusing 3800 Shimadzu in the range of (4000-400)cm^{-1}. Electronic spectra were obtained usingUV-160 Shimadzu spectrophotometer at 25 C for 10^{-3} M solution DMSO with1.000±0.001cm matched quartzcell.Molar Conductivity was measured at 25 C for10^{-3}M solution of DMSO by using PhilipsPW..Digital.Micro elemental analysis (C.H.N.S) were performed using AcrloErba1106elementalanalyzer. Magnetic susceptibility measurements were Obtained by balance magnetic susceptibilityby modelMSB-MKI.Metal contents of the complexes were determine di by atomic absorption technique by using Shimadzu (AA680G).

Preparation of ligand(CPC)

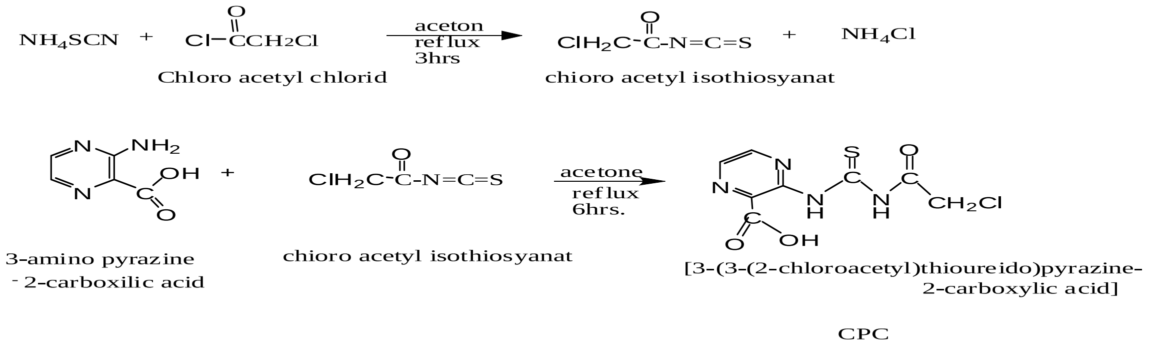
The ligand was prepared by twosteps(scheme-)

(A)- Preparation of the(Chloro acetyl isothiocyanate)[10]

Mixture of chloroacetyl chloride(2.05ml, 26mmol)and ammonium thiocyanate (2g,26mmol) in (25ml) of acetone was stirred under refluxed for 3 hrs and then filtered ,the filtrate was used for further reaction .

(B)Preparation of [3-(3-(2-chloroacetyl)thioureido)pyrazine-2-carboxylic acid] (CPC)

(3.616g,26mmol) of 3-amino pyrazine-2-Carboxilic Acid in(20ml) acetone wasrapidly added to Chloro acetyl isothiocyanate and maintainingreflux.Afterrefluxing for 6hrs,theresultingsolidwas collected, washed with acetone and recrystallization from ethanol,yield(80%), (m.p=250-52)°C,C%found (34.58)calc.(34.97), H% found (2.61) calc.(2.56), N% found (20.45) calc.(20.40),S%found(12.12)calc. (11.67) Scheme. (1).



Scheme. (1): The synthesis of ligand (CPC)

Synthesis of metal complexes

(0.67g ,2mmole) of ligand (CPC) was dissolved in 20 ml of ethanol containing (0.12g , 2mmole) of KOH, then the solution of following metal salts $MnCl_2$.$4H_2O$ (0.2g,1mmole),$CoCl_2.6H_2O$ (0.24g,1mmole) $NiCl_2.6H_2O$ (0.24g,1mmole),$CuCl_2$.$2H_2O$ (0.2g ,1mmole), $ZnCl_2$ (0.14g,1mmole),$CdCl_2$ .H_2O (0.2g ,1mmole),and $HgCl_2$(0.3g ,mmole) 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

(3-5hours).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

Ligand (CPC)

The FT-IR spectrum of the free ligand (CPC) ,Fig.(1) showed bands due to υ(OH), υ(NH) amide,υ(C=O) (amidic) and υ(C=S) which were observed at (3002)cm^{-1},(3251) cm^{-1} (1600)cm^{-1}and(1211)cm^{1} ,respectively . While another absorption band appeared at (1705)cm^{-1} could be explained as υ(COO)$_{asym}$ were the υ(COO)$_{sym}$ was noticed (1396)cm^{-1} [11]. spectral data of the free ligand were listed in table (2).

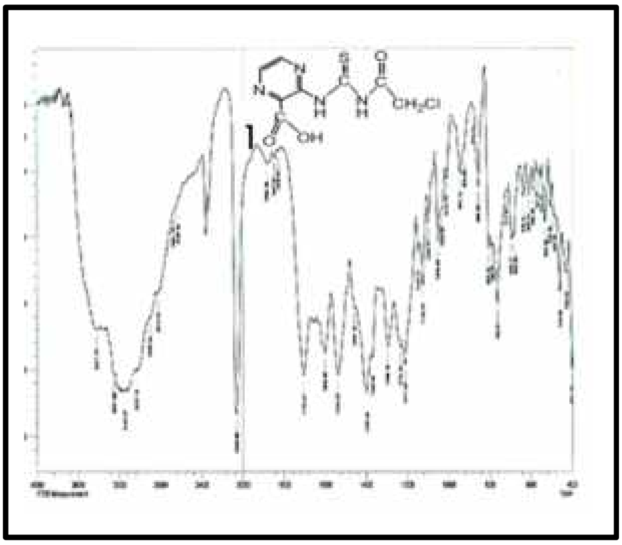


Fig. (1): Infrared spectrum of ligand (CPC)

The UV-Vis spectrum of the free ligand (CPC).Fig.(2) exhibits ahigh intense bsorption peak at (36363) cm^{-1} ,(25575) [12]. which may be attributed to electronic transition type π ⟶ π,*n ⟶ π*respectively.The data of electronic spectrum of the free ligand (CPC) were listed in table (3).

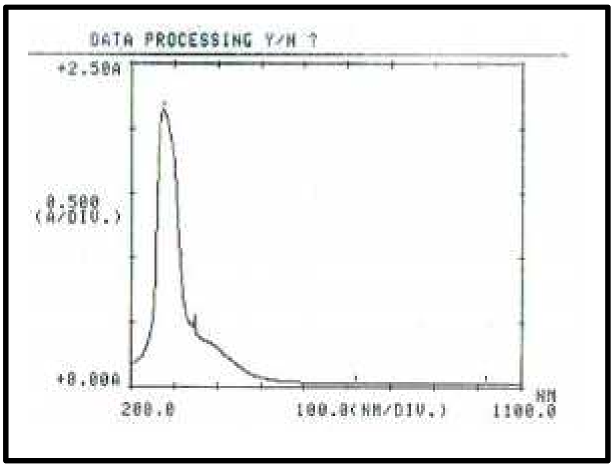


Fig . (2) : Electronic spectrum of ligand (CPC)

The ^{1}H-NMR spectrum of free ligand (CPC),Fig .(3) which was recorded in DMSO-d^{6} solvent showed the following signalsi: single at δ(4.48-4.50) ppm for (2H,CH$_2$Cl),singlet Peak at(2.5) ppm for DMSO, multiplet peak at δ(6.92-7.26) ppm for aromatic protons), singlet peak at δ (7.93) ppm refers to (1H, NH sec , amine), signals peak at δ (8.84) ppm, for (1H ,NH-CS). signals peak at δ(11.56) ppm for(1H,COOH) [13].

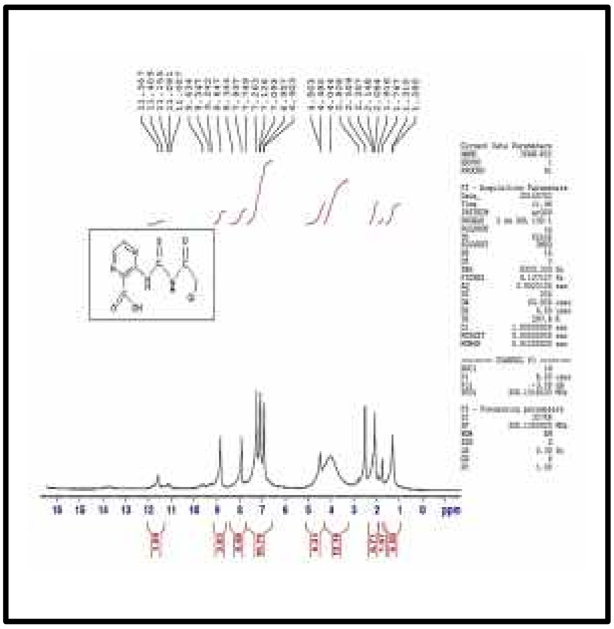


Fig.(3) :^{1}HNMR spectrum of ligand (CPC)

^{13}C-NMR spectrum of the free ligand(CPC) in DMSO-d$_6$,Fig.(4)showed for following singals: singals at δ(38.68-40.67) ppm for DMSOandCH$_2$Cl signal at δ(114.92-149.32) ppm for ppm for aromatic carbons, singals

atδ(164.92)ppmfor(COOH),signal
aδ(171.84)ppm(C=Osec.amid),signal
atδ(180) ppm for(C=S) .

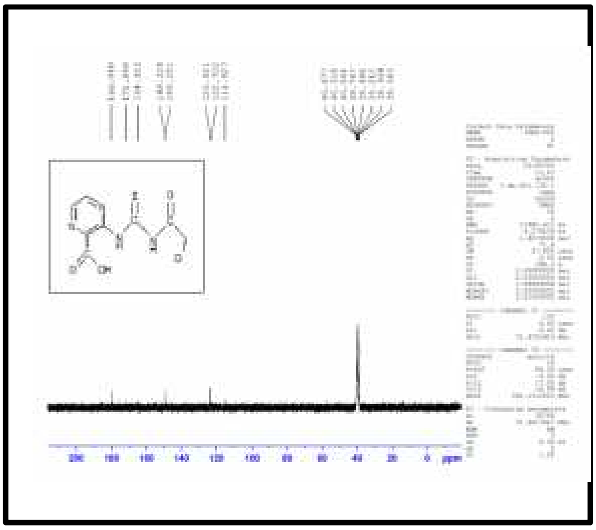


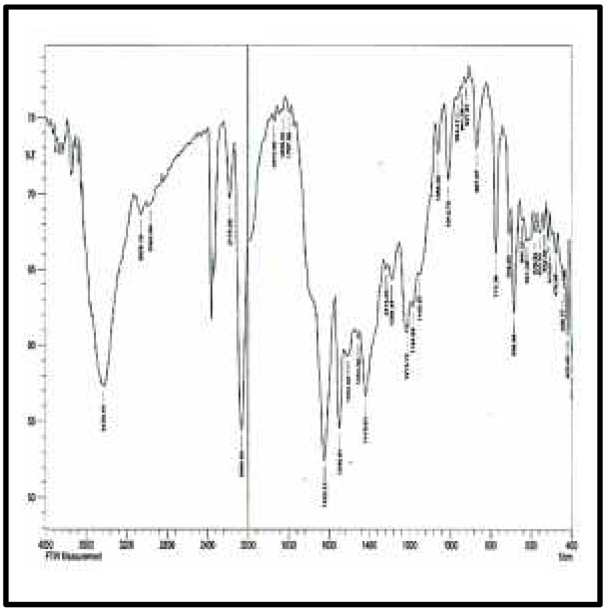
Fig. (4) :^{13}CNMR spectrum of ligand (CPC)

Complexes of the ligand(CPC)

The solid complexes soluble in some common solvent such as dimethyl formamide , di methyl 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 to be non- electrolyte .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.(5)belonging to the stretching vibration of band υ(NH)of the amine group in the range between (3429-3352) cm^{-1} shifted to higher frequencies by(177-101)cm^{-1}suggesting of the possibility of the coordination of ligand through the nitrogen atom at the amine group[14-17].absorption assigned for υ(COO)sym was noticed at the range (1419-1384) cm^{-1} shifted to higher frequencies by (50-35)cm^{1}whilethebandcausedbyυ$(COO)_{Asym}$ appeared between (1670-1635) cm^{-1}shifted to lower frequencies by (70-35)cm^{-1} which indicates to the coordination of carboxylic group to the central ion [18]. the stretching vibration band carbonyl group υ (C=O) and υ (C=S) either show no change or very little in their frequencies (1620-1597)cm^{-1} and (1238-1215)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 (447-405) Cm^{-1} and (495-447) Cm^{-1} respectively Table (2) describe the important bands and assignment for free ligand (CPC) and its complexes .



Fig(5) : Infrared spectrum of $[Mn(CPC)_2]$

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).Ni(II),Mn(II), Co(II) andCu (II),complexes exhibit μeff (5.78,4.87, 2.94,1.76) B.M respectivelywhich can be anormal values for high spinTetrahedral complexes .[20]

Electronic spectra for complexes

-$[Mn(CPC)_2]$ d^5

The Brown complex of Mn (II) shows band at(37037)cm^{-1} due to ligand field and other bands at (30674)cm^{-1} and (9852) cm^{-1} which are caused by the electronic transfer which are caused by the electronic transfer $^6A_1 \longrightarrow {}^4T_{1(P)}and^6A_1 \longrightarrow {}^4T_{1(G)}$

$^6A_1 \longrightarrow {}^4T_{1(G)}$ respectively , suggesting Tetrahedral geometry aroundiMn(II) ion [21].

-[Co(CPC)$_2$] d^7

The spectrum of the Green complex gave four bands at (36363) cm^{-1} ,(28901) cm^{-1} , (14925)cm^{-1} and (10799) cm^{-1} attributed to (L.F),C.T mixed with $^4A_2 \longrightarrow {}^4T_{1\,(P)}$, $^4A_2 \longrightarrow {}^4T_{1\ (F)}$ and $^4A_2 \longrightarrow {}^4T_{2\ (F)}$ respectively and the racah inter electronic repulsion parameter (B$^-$) was found to be (761) cm^{-1} , from the relation $\beta = B^- / B^0$, was found to be equal (0.78),these parameter are accepted to Co (II)Tertahedral complex [22].

-[Ni(CPC)$_2$] d^8

The spectrum of Blue complex of Ni (II)has revealed the following electroni ctransfer (L.F),C.T mixed with$^3T_{1(F)} \longrightarrow {}^3T_{1\,(P)}$. $^3T_{1(F)} \longrightarrow {}^3A_2and^3T_{1(F)} \longrightarrow {}^3T_{2(F)}$ transition at(36231)cm-^{1},(28735) cm^{-1},(23255)cm^{-1}and(13717)cm-^{1}respectively, the(B$^-$) value is found to be (722)cm-1 ,while β was equal to (0.69) these are the characteristics for Tetrahedral complexes of Ni (II)[23].

-[Cu (CPC)$_2$] d^9

The spectrum of Deep Green complex of Cu (II)Fig . (6) shows two bands at(37037) cm^{-1},(11111)cm^{-1} causedto (L.F),$^2B_1g \longrightarrow {}^2A_1g$ transition respectively ,which was agood agreement for square Planer complex for Cu(II) ion[24].

agreement for square Planer complex for Cu(II) ion [24].

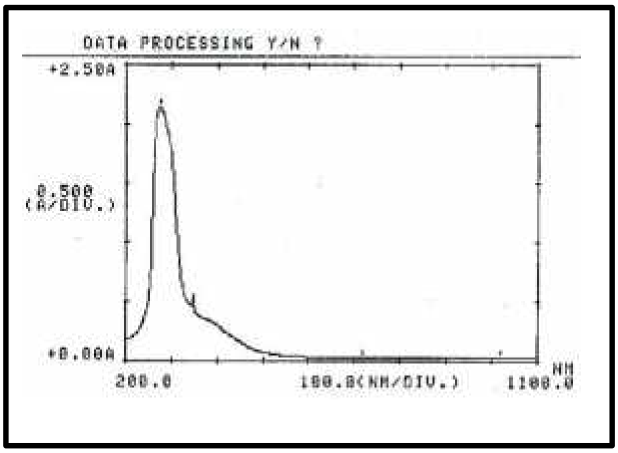


Fig.(6):Electronic spectrum of [Mn (CPC)$_2$]

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

Compound	**M.wt (gm/mole)**	**Color**	**M.P(C) or dec.**	**M% Calculation (Found)**	**Molar Cond. Ohm^{-1}cm2mol^{-1} in DMSO**	**µeff (B.M)**
$C_8N_4O_3H_7ClS$ (CPC)	274.68	Brown	250-252	-	-	-
[Mn$(CPC)_2$]	602.36	Brown	260 (dec)	8.92 (9.13)	17.2	5.78
[Co $(CPC)_2$]	606.23	Blue	285(dec)	9.32 (9.72)	7.71	4.87
[Ni$(CPC)_2$]	606.05	Green	264	9.29 (9.68)	14.2	2.94
[Cu$(CPC)_2$]	610.90	deep Green	265	10.66 (10.32)	12.	1.76
[Zn$(CPC)_2$]	612.76	Brown	290(dec)	10.89 (10.66)	13.4	0
[Cd$(CPC)_2$]	659.76	Brown	293	17.27 (17.03)	18	0
[Hg$(CPC)_2$]	747.94	Brown	270 (dec)	27.01 (26.82)	8.5	0

dec.= decomposition

Table(2)The characteristic bands of infrared spectra of ligand and complexes

Compound	**υ(N-H)** **υ(O─H)**	**υ(COO)** **smm**	**υ(COO)** **a smm**	**υ(C=O)**	^{3}**(C=S)**	**υ(M-N)**	**υ (M─O)**
Ligand (CPC)	3251 (m) 3002 (s)	1396 (s)	1705 (s)	1600(s)	1211 (s)	—	—
[Mn(CPC)$_2$]	3429 (S)	1419 (s)	1670 (s)	1620 (m)	1215 (s)	478 (w)	420 (w)
[Co(CPC)$_2$]	3383 (b)	1384 (m)	1650 (s)	1608 (m)	1230 (s)	447 (m)	420 (w)
[Ni(CPC)$_2$]	3388 (m)	1400 (m)	1652 (s)	1616 (m)	1226 (s)	491 (w)	420 (w)
[Cu(CPC)$_2$]	3410 (m)	1419 (m)	1639 (s)	1620 (m)	1230 (s)	495 (w)	405 (w)
[Zn(CPC)$_2$]	3352 (m)	1415 (m)	1651 (m)	1597 (m)	1238 (s)	482(m)	435 (w)
[Cd(CPC)$_2$]	3410 (m)	1384 (m)	1635 (m)	1616 (s)	1226 (s)	474 (m)	447 (w)
[Hg(CPC)$_2$]	3383 (m)	1411 (m)	1660 (m)	1620 (s)	1222 (s)	482 (w)	422 (w)

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

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

Compounds	^{a}(nm)	υ-(cm^{-1})	ABC	Emax molar^{-1}cm^{-1}	Transitions
Ligand(CPC)	275	36363	2.144	2144	$\pi \rightarrow \pi^*$
	391	25575	0.685	685	$n \rightarrow \pi^*$
[Mn(CPC)$_2$]	270	37037	1.187	1187	L.F
	326	30674	1.629	1626	${}^6A_1 \rightarrow {}^4T_{1\,(p)}$
	1015	9852	0.018	18	${}^6A_1 \rightarrow {}^4T_{1\,(G)}$
[Co (CPC)$_2$]	275	36363	1.724	1724	L.F
	346	28901	1.266	1266	C.T mixed with ${}^4A_{2\,(F)} \rightarrow {}^4T_{1\,(P)}$
	670	14925	0.018	18	${}^4A_{2\,(F)} \rightarrow {}^4T_{1\,(p)}$
	926	10799	0.015	15	${}^4A_{2\,(F)} \rightarrow {}^4T_{1\,(F)}$
[Ni(CPC)$_2$]	276	36231	2.193	2193	L.F
	346	28735	1.494	1494	C.T mixed with ${}^3T_{1\,(F)} \rightarrow {}^3T_{1\,(P)}$
	670	23255	0.158	158	$3T_{1\,(F)} \rightarrow {}^3A_{2\,(F)}$
	926	13717	0.032	32	${}^3T_{1\,(F)} \rightarrow {}^3T_{2\,(F)}$
[Cu(CPC)$_2$]	271	37037	1.480	1480	L.F
	900	11111	0.018	18	${}^2B_1g \rightarrow {}^2A_2g$
[Zn (CPC)$_2$]	278	35971	2.119	2119	L.F
	345	28985	1.538	1538	C.T
[Cd(CPC)$_2$]	277	36101	2.157	2157	L.F
	345	28985	1.666	1666	C.T
[Hg (CPC)$_2$]	271	36900	2.823	2823	L.F
	323	30959	2.084	2084	C.T

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

-The complexes of$[Zn(CPC)_2]$,$[Cd(CPC)_2]$ and $[Hg(CPC)_2]$

Show only charge transfer of (M→L) in range (35971-28985)cm^{-1} [25]. All transition with their assignments are summarized in Tabl-e (3).Suggested structures for complexes on the basis of molar conductivity, magnetic moment, spectroscopic studies (FT-IR,UV-Vis and atomic absorption) and (^{1}H-^{13}CNMR for ligand(CPC) only)for the ligand and all prepared complexes, we suggested that the ligand (CPC) 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 carboxylic group and nitrogen atom of amino group, suggesting tetrahedral geometry for prepared complexes except copper complex has square planer geometry.

Conclusions

The new ligand in this work has been readily prepared by reaction from Chloro acetyl isothiocyanate with 3-amino pyrazine-2-Carboxilic.The ligand was characterized characterized by elemental micro analysis C.H.N.S , FT-IR , UV-Vis and ^{1}H,^{13}C-NMR spectra. The metal complexes of this ligand were prepared and characterized by FT-IR, UV-Vis spectra, conductivity measurements, magnetic susceptibility and atomic absorption, the proposed geometrical structure for complexes were tetrahedral geometry except copper complex has square planer.

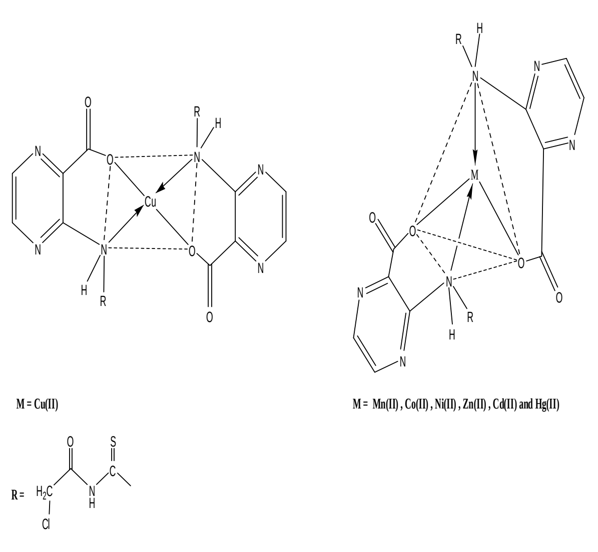


Figure. (7):The proposed chemical structure formula of the complexes.

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تحضير ودراسة طيفيه لبعض المعقدات الفلزية مع
[3-(3-(2-كلورواسيتايل)ثايويوريا)بايرازين-2- كاربوكسيك اسيد]

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الخلاصة

حضر الليكاند الجديد[3-(3-(2-كلورو اسيتايل) ثايويوريا)بايرازين -2- كاربوكسيك اسيد](CPC)وذلك من مفاعله (كلورو اسيتايل ايزوثايوسيانات) مع 3-امينوبايرازين 2- كاربوكسيلك اسيد 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 (Hg, Cd , Zn, Cu , Ni, Co , Mn) مع الليكاند (CPC) وشخصت المعقدات المحضرة باستعمال الأشعة تحت الحمراء والأشعة فوق البنفسجية - المرئية والتوصيلية المولارية والحساسية المغناطيسية والامتصاص الذري ونستنتج من الدراسات والتشخيصات إن المعقدات لها شكل رباعي السطوح ماعدا النحاس الذي اقترح له الشكل المربع المستوي حول الايون الفلزي مع الليكاند (CPC) ثنائي السن.

الكلمات المفتاحية :, 3-امينوبايرازين -2- كاربوكسيلك اسيد,استيل ايزوثايوسيانات, معقدات

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