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Synthesis and Spectroscopic Studies of some Divalent Metal Ion Complexes of 3-(4-hydroxyphenyl)-2-(3-(4-nitrobenzoyl) thioureido) propanoic acid

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Abstract:

3-(4-hydroxyphenyl)-2-(3-(4-nitrobenzoyl) thioureido) propanoic acid (HNP) a new ligand was synthesized by reaction of Tyrosine with (4-Nitrobenzoyl isothiocyanate) by using acetone as a solvent. The prepared ligand (HNP) has been characterized by elemental analysis (CHNS), infrared (FT-IR), electronic spectral (Ultraviolet visible) and(^{1}H,^{13}C-Nuclear Magnetic Resonance) spectra. Some Divalent metal ion complexes of (HNP) were prepared and spectroscopic studies by Fourier transform infrared (FTIR), electronic spectral(UV-Vis), molar conductance, magnetic susceptibility and atomic absorption. The results measured showed the formula of six prepared complexes were [M $(HNP)_2$] (M^{+2} = Manganese, Cobalt, Nickel, Znic, Cadmium and Mercury),from the obtained data tetrahedral structure suggested for all complexes, except Copper complex has a square planer geometry.

Key words: Tyrosine, Divalent Metal Ion, 4-Nitrobenzoyl isothiocyanate.

Introduction:

Both functional groups amine ($-NH_2$) and carboxyl (COOH), that amino acid contains, give the amino acid a biological important organic compounds , along with a side-chain(R group)specific to each amino acid, because of their biological importance. Amino acids are necessary in nutrition and commonly used in dietary supplements , compost and food technology. Industrial uses include the production of drugs, biodegradable plastics, and chiral catalysts [1]. Tyrosine is the one of standard amino acid; it is aromatic and anon-essential amino acid with a polar side group.

Tyrosine is a precursor to neurotransmitters and increases plasma neurotransmitter levels (particularly dopamine and norepinephrine) [2] but has little if any effect on mood[3]. The effect on mood is more eminent in humans subjected to stressful conditions ,L-tyrosine and its derivatives (L-DOPA, melanin, phenylpropanoids, and others) are used in pharmaceuticals, nutritional supplements , and food additives. Yang Z. and coworkers [4] was prepared a copper (II) complex with tyrosine of formulation[Cu-Phen-Tyr](H_2O)](ClO_4) (Phen = 1,10-phenanthroline, L: -Tyr =

L: -tyrosine) and also Each Abdul Wajid* and R. B. Mohod [5]were preparing new Schiff Base Complexes of Mn(II), Co(II), Ni(II), Cr(III), Cu(II), Zn(II) and Cd(II)from a new ligand [DHPEAHP] with formulation [M(DHPEAHP) (H_2O)] when [DHPEAHP]= [1-(2,4-Dihydroxy-phenyl)-ethanone-(S)-Alpha-amino-4-hydroxybenzene propanoic acid]

The aim of this work is to synthesis a new ligand [3-(4-hydroxyphenyl)-2-(3-(4-nitrobenzoyl) thioureido) propanoic acid] (HNP),and it's metal complexes with Mn^{+2}, Co^{+2}, Ni^{+2}, Cu^{+2} , Zn^{+2} Cd^{+2} and Hg^{+2}.

Materials and Methods:

Chemicals

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

Instruments

- ^1H and ^{13}C–NMR were recorded using Ultra Shield 300MH_z Switzerland at University of Al al-Bayt, Jordan.
- Melting point was recorded by using melting point meter KRUSS (A, KRUSS OPTRONIC).
- FT-IRspectra were recorded as FT-IR using 3800Shimadzu in the range of (4000-400) $cm^{-1}$by using (KBr) disc.
- Electronic spectra were obtained using (UV-160 Shimadzu) spectrophotometer at 25℃ for 10^{-3}M solution DMSO.
- Molar Conductivity was measured at 25℃ for 10^{-3}M by using PhilipsPW.
- Digital micro elemental analysis (C.H.N.S) were performed using AcrloErba 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 in Ibn-Sina Company by using Shimadzu (AA680G).

Preparation of ligand(HNP)

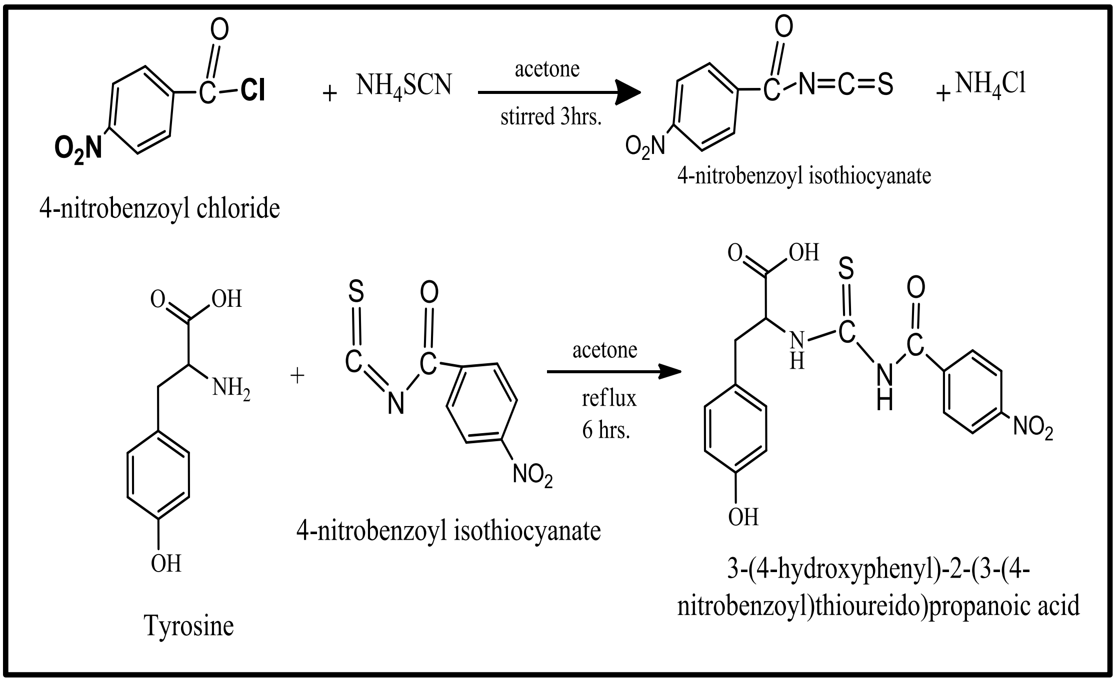
It contains two steps:

1-Synthesis of compound (4-nitrobenzoyl isothiocyanate) [6]

Mixture of (2g, 26mmol) of ammonium thiocyanate with (3.21g, 26mmol) of 4-nitrobenzoyl chloride in (25 ml) of acetone was stirred for (3 hrs.) and then filtered.

2-Synthesis of Lignd 3-(4-hydroxy phenyl)-2-(3-(4-nitrobenzoyl) thioureido) propanoic acid (HNP)

A solution of (4.71g,26mmol) tyrosine in (20ml) acetone was rapidly added to the intermediate and reflux. After refluxing for(6 hrs.), the resulting solid was collected, washed with acetone and recrystallized with ethanol as in Scheme (1) Yield (80%), (m.p=158-160)˚C, C% found (52.33) calc.(52.44), H% found (3.72), calc.(3.88),N% found (11.41), calc.(10.59),S% found (8.66), calc.(8.23).



Scheme (1) Preparation of (HNP)

Synthesis ligand (HNP) complexes:

Synthesis of the [Mn(HNP)$_2$] complex

A solution of (0.196 g,1mmol) $MnCl_2.4H_2O$ in (10ml) ethanol was added by drop wise to solution of the ligand (HNP^-K^+) that prepared from (0.78g, 2mmol) (HNP) in (10ml) ethanol containing (0.12g , 2mmole) of KOH.

At room temperature, the mixture was stirred for 3hours. The precipitate was collected by filtration, washed with mixture of water: ethanol(1:1)then dried in an oven at(50˚C).

Synthesis of [Co(HNP)$_2$], [Ni(HNP)$_2$] , [Cu(HNP)$_2$], [Zn(HNP)$_2$], [Cd(HNP)$_2$] and [Hg(HNP)$_2$] complexes

A similar method to that above-mentioned for preparation of [Mn(HNP)$_2$] complex was used to prepare the complexes by using (1mmol) (0.13gm, 0.24gm, 0.17gm, 0.136gm, 0.2gm & 0.27gm) for ($CoCl_2.6H_2O$, $NiCl_2.6H_2O$, $CuCl_2.2H_2O$, $ZnCl_2$, $CdCl_2.H_2O$ and $HgCl_2$) respectively. Some physical properties of the prepared complexes are showed in Table(1)

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

Compound	M.wt (gm/mole)	Color	M.P(°C) Or dec .	M% Calculation (Found)	Molar Cond. Ohm $^{-1}$cm^{2}mol^{-1} In DMSO	μ_{eff} (B.M)
Lignd(HNP) $C_{17}H_{15}O_6N_3S$	389.38	Brown	158-160	----	----	—
[Mn(HNP)$_2$]	831.69	Light Green	168-170	6.61 (5.89)	14.5	5.93
[Co(HNP)$_2$]	835.68	Green	166-168	7.05 (6.91)	11.1	4.91
[Ni(HNP)$_2$]	835.44	greenish yellow	232-233 Dec.	7.03 (6.80)	15.4	2.99
[Cu(HNP)$_2$]	840.30	Orang	220-222	7.56 (7.01)	13.4	1.78
[Zn(HNP)$_2$]	842.13	Yellow	230-231 Dec.	7.76 (8.02)	15.8	0.00
[Cd(HNP)$_2$]	889.16	Yellow	200-202	12.64 (12.35)	14.0	0.00
[Hg(HNP)$_2$]	977.34	Deep Yellow	210-212	20.52 (21.23)	11.8	0.00

Dec.= decompose

Results and Discussion:

1- Ligand (HNP)

The(^{1}H-NMR)spectrum of the ligand(HNP) Figur(1) in DMSO shows the following signals: doublet peak at δ(2.2)ppm for(1H,NH Sec amine), singlet peak at δ(2.5)ppm for DMSO, singles peaks at δ(2.71 – 3.21)ppm for (2H,CH2 and 1H,CH),singlet peak at δ (5.03 – 5.04)ppm for (1H, OH phenolic),doublet doublet peaks at δ(6.65–8.68)ppm for (8H,aromatic protons), singlet peak at δ(9.66)ppm for (1H, NH sec, amide), singlet peak at δ(11.10)ppm for (1H, COOH)

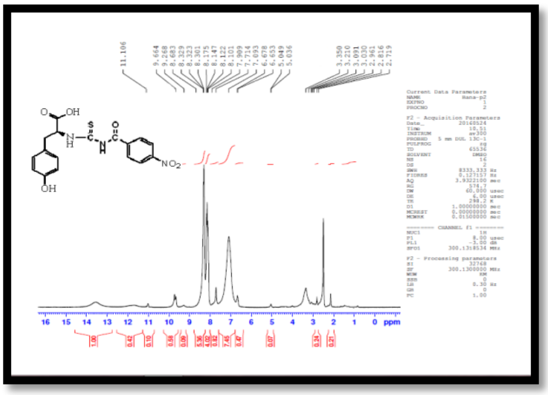


Fig.(1) : ^{1}H-NMR spectrum of ligand (HNP)

The(^{13}C-NMR) spectrum of the ligand (HNP) Figur(2) shows the following signals: signal at δ (38.63 – 40.30) ppm for DMSO, signal at δ (115.18 – 149.97)ppm for aromatic carbons, signal at δ (165.77)ppm for (C=O sec. amid), signal at δ (166.52)ppm for (C = O carboxylic), signal at δ (171.32)ppm for (C=S)[7-8]

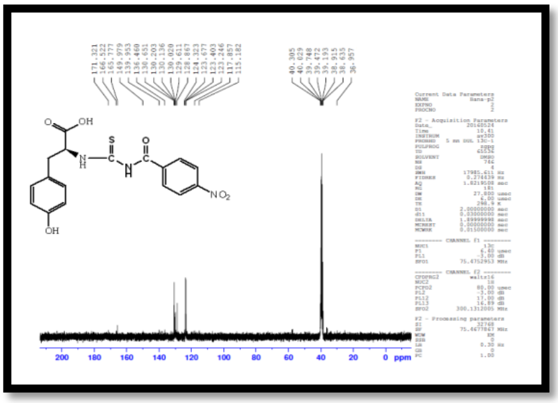
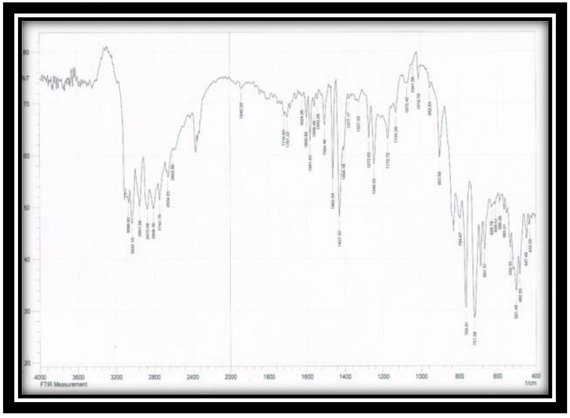


Fig.(2): ^{13}C-NMR spectrum of ligand (HNP)

The(FT-IR) spectrum of the free ligand(HNP), Figure(3),shows broad band at (2951,3066) cm^{-1}due to υ(OH, NH_2).While other absorption bands at (1701) cm^{-1} ,(1327) cm^{-1} ,(1600) cm^{-1} ,(1246)cm^{-1} due to $\upsilon(COO_{asy})$, $\upsilon(COO_{sym})$, υ(C=O amidic)and υ(C=S) respectively[9-10].The FT-IR spectral data of the free ligand(HNP) are listed in Table(2).



Fig(3): Infrared spectrum of ligand (HNP)

The (UV-Vis) spectrum of the free ligand(HNP), Figure(4) gives a high intense absorption peak at(36764) cm^{-1} which may be attributed to electronic transition type $\pi\rightarrow\pi^*$ [11].The data of electronic spectrum of the free ligand (HNP)is registered in Table (3).

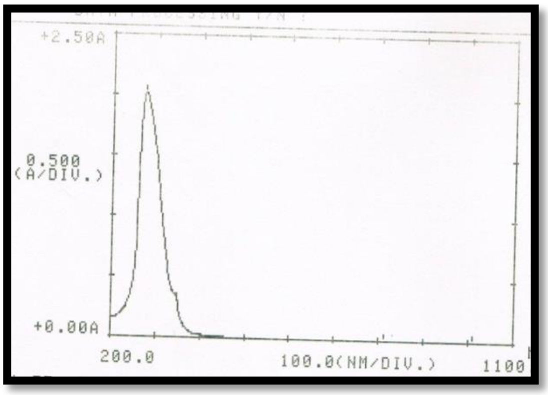


Fig.(4): U.V spectrum of ligand (HNP)

2- Complexes of the ligand (HNP)

The precipitate complexes soluble in some common solvents are "dimethyl formamide","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 to be non-electrolytic nature [12].The atomic absorption measurements for all complexes gave approximated values when it is compared with theoretical values. Physical properties of the ligand(HNP) and its complexes shown in Table (1).

FT-IR Spectra

These spectra exhibited marked difference between bands Figure(5) shows Infrared spectrum of complex[Ni $(HNP)_2$]. The stretching vibration of υ(NH) between(3217-3116)cm^{-1}shifted higher frequencies by(175-50)cm^{-1} suggesting of the coordination of ligand through the nitrogen atom from υ(N-H) group[13] while the band at(1701)cm^{-1} was assigned to the stretching of $\upsilon(COO)_{asym}$ group, in the spectra of complexes. This band has been found in the range between(1666-1689)cm^{-1} ,so the band was shifted to lower frequencies by(35-11)cm^{-1} from the free ligand; the band at(1327)cm^{-1} was assigned to the stretching of $\upsilon(COO)_{sym}$ group[14] in the spectra of complexes. This band has been found in the range between (1411-1435) cm^{-1} shifted to higher frequencies by (84-108) cm^{-1}. Which indicates the coordination of the carboxylic group to the central ion as a mono dentate. The stretching vibration band $\upsilon(C=O)_{amid}$ and υ(C=S) either show no change or very little in their frequencies (1620-1604)cm^{-1},(1230-1280)cm^{-1},respectively; therefor, indicating do not coordinate to the metal ion. Metal-oxygen and metal-nitrogen were confirmed by the presence of the stretching tremor of υ(M-N) and υ(M-O) around (443-466)cm^{-1} and (428-401)cm^{-1} respectively[15].Table (2) describes the important bands and assignment for all prepared complexes.

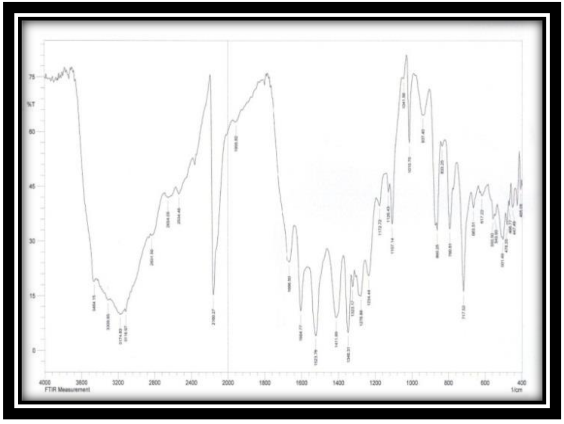


Fig.5: Infrared spectrum of complex [Ni $(HNP)_2$]

Table (2): The characteristic infrared band for free ligand (HNP) and its metal complexes

Compounds	υ(O ─H) υ(N─ H)	υ(COO) asym	υ(COO) sym	Δυ (COO)	υ(C=O) Amide	υ (C=S)	υ (M-O)	υ (M-N)
Ligand (HNP)	$2951_{(m)}$ $3066_{(m)}$	$1701_{(m)}$	$1327_{(w)}$	426	$1600_{(m)}$	$1246_{(s)}$	---	---
$[Mn(HNP)_2]$	$3155_{(b)}$	$1666_{(s)}$	$1411_{(s)}$	255	$1604_{(s)}$	$1238_{(w)}$	$420_{(m)}$	$459_{(m)}$
$[Co(HNP)_2]$	$3159_{(b)}$	$1670_{(s)}$	$1411_{(s)}$	259	$1604_{(s)}$	$1242_{(m)}$	$405_{(m)}$	$455_{(m)}$
$[Ni(HNP)_2]$	$3174_{(b)}$	$1666_{(s)}$	$1411_{(s)}$	255	$1604_{(m)}$	$1234_{(m)}$	$405_{(m)}$	$466_{(m)}$
$[Cu(HNP)_2]$	$3116_{(b)}$	$1670_{(s)}$	$1415_{(s)}$	255	$1604_{(s)}$	1250(s)	$405_{(m)}$	$459_{(m)}$
$[Zn(HNP)_2]$	$3217_{(b)}$	$1689_{(s)}$	$1435_{(s)}$	254	$1620_{(s)}$	$1257_{(w)}$	$405_{(m)}$	$459_{(m)}$
$[Cd(HNP)_2]$	$3174_{(b)}$	$1680_{(s)}$	$1415_{(s)}$	265	$1604_{(s)}$	$1250_{(w)}$	$408_{(m)}$	$443_{(w)}$
$[Hg(HNP)_2]$	$3174_{(b)}$	$1669_{(s)}$	$1416_{(s)}$	253	$1604_{(s)}$	$1230_{(w)}$	$401_{(m)}$	$443_{(m)}$

Electronic spectra for complexes and Magnetic moment

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

The Light green complex of Mn(II) as shown in Figure (6)gives band at (36101) cm^{-1} due to (L.F) and other bands at (28985) cm^{-1} and (112300)cm^{-1} which are caused by the electronic transfer $^6A_1 \longrightarrow {}^4T_2$ and $^6A_1 \longrightarrow {}^4T_1$ respectively. The value of measured μeff for the Mn(II)is(5.93),We propose Tetrahedral geometry around Mn^{+2} [16-17].

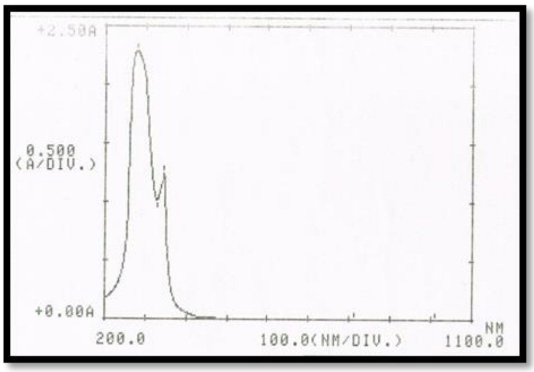


Fig.(6): U.V spectrum of complex $[Mn(HNP)_2]$

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

The spectrum of the green complex gave four bands at (35971) cm^{-1},(25641)cm^{-1}, (12722) cm^{-1}& (10373) cm^{-1} attributed to (L.F) with $^4A_2 \longrightarrow {}^4T_1$, $^4A_2 \longrightarrow {}^4T_1$ and $^4A_2 \longrightarrow {}^4T_2$ respectively and Racah inter electronic repulsion parameter (B^-)was found to be (483) cm^{-1}, from the relation $\beta = B^-/B^0$, was found to equal (0.50). The value of measured μeff for the Co(II) is (4.91) B.M. these parameters are accepted to Co^{+2} Tetrahedral complex [18].

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

The spectrum of greenish yellow complex of Ni(II)has revealed the following electronic transfer(L.F) with $^3T_{1\ (F)} \longrightarrow {}^3T_{1(P)}$, $^3T_1 \longrightarrow {}^3A_1$, and $^3T_1 \longrightarrow {}^3T_2$, transition at (36363)$cm^{-1}$, (27777)$cm^{-1}$, (13623)$cm^{-1}$ and (9891) cm^{-1} respectively. The(B^-) value is found to be (781.8)cm^{-1}, while β was equal to (0.751).These are the characteristics for Tetrahedral complexes of Ni^{+2} [19] The value of measured μeff for the Ni(II) is (2.99) B.M.

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

The spectrum of Orang complex of Cu(II) Fig.(6) shows two bands at (36630) cm^{-1}, (12300) cm^{-1} and (11560) cm^{-1} caused to (L.F.)$^2B_1g \longrightarrow {}^2A_1g$,$^2B_1g \longrightarrow {}^2B_2g$ transition respectively and the value of measured μeff for Cu(II) showed μeff(1.78) B.M, which was a good agreement for Square planer complex for Cu^{+2} [20-21].

-The complexes of $[Zn(HNP)_2]$, $[Cd(HNP)_2]$ and **$[Hg(HNP)_2]$** show only(C.T) and (L.F)of (M→L) in range (36764-18939) cm^{-1}[22]. All transitions with their assignments are summarized in Table (3)and the values of measured magnetic susceptibility and (μeff) for the Mn(II), Co(II), Ni(II), Cu(II) complexes were registered in Table(1)

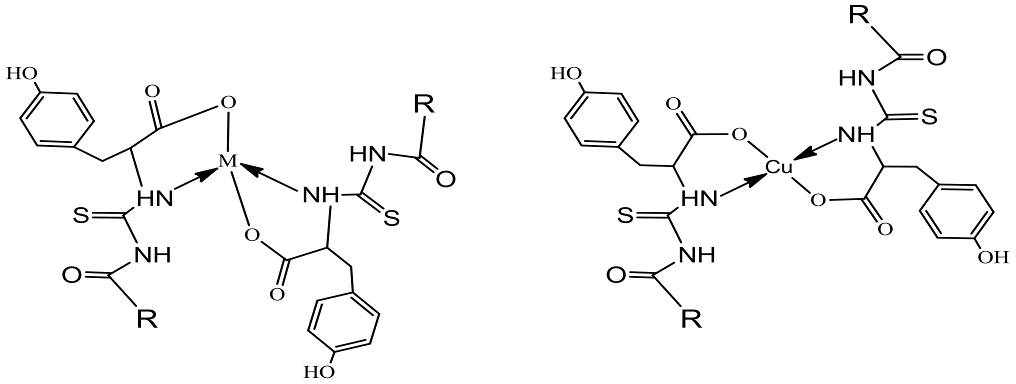
Table (3) Electronic spectral data of ligand (HNP) and its complexes in DMSO solvent

compounds	λ(nm)	$\bar{\upsilon}$(cm^{-1})	ABC	ε_{max} molar^{-1}cm^{-1}	Transitions
Ligand (HNP)	272	36764	2.016	2016	$\pi\rightarrow\pi^*$
[Mn(HNP)$_2$]	277 345 813	36101 28985 12300	2.288 1.248 0.020	2288 1248 20	L.F. $^6A_1 \rightarrow {}^4T_2$ $^6A_1 \rightarrow {}^4T_1$
[Co(HNP)$_2$]	278 390 786 964	35971 25641 12722 10373	2.311 0.990 0.020 0.018	2311 990 20 18	L.F. $^4A_2\rightarrow {}^4T_1$ $^4A_2\rightarrow {}^4T_1$ $^4A_2\rightarrow {}^4T_2$
[Ni(HNP)$_2$]	275 360 734 1011	36363 27777 13623 9891	2.213 0.755 0.025 0.018	2213 755 25 18	L.F. $^3T_1$ (F) $\rightarrow {}^3T_1$ (P) $^3T_1 \rightarrow {}^3A_2$ $^3T_1 \rightarrow {}^3T_2$
[Cu(HNP)$_2$]	273 813 865	36630 12300 11560	2.056 0.028 0.018	2056 28 18	L.F. $^2B_1g \rightarrow {}^2A_1g$ $^2B_1 g \rightarrow {}^2B_2g$
[Zn(HNP)$_2$]	275 538	36363 18939	2.114 0.075	2114 75	L.F C.T
[Cd(HNP)$_2$]	272 345	36764 28985	1.957 0.469	1957 469	L.F C.T
[Hg(HNP)$_2$]	273 352	36630 28409	2.069 0.250	2069 250	L.F C.T

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

Conclusions:

As demonstrated by looking at the research and depending on the results obtained molar conductivity, magnetic moment, spectroscopic studies (FT-IR,UV-Vis and atomic absorption)for the ligand (HNP) and all prepared complexes add to (^{1}H-^{13}C NMR)only for ligand, the new prepared ligand (NHP) action as bi dentate on coordination with central ion Mn(II), Co (II), Ni(II), Cu(II), Zn(II), Cd(II) & Hg(II) via oxygen atom of (COO) carboxylic group and nitrogen atom of (NH) group, So we propose Tetrahedral geometry around metal ions for all prepared complexes, except Copper complex that has a Square planer geometry as shown in Figure(7).



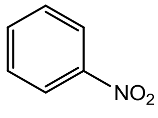
M^{+2}= Mn , Co, Ni, Zn, Cd, Hg R= 

Fig 7: The proposed chemical structure formula of the complexes

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تحضير ودراسة طيفية لبعض معقدات الفلزات ثنائية التكافؤ مع 3-(4-هايدروكسي فنل)-2-(3-(4-نايتروبنزويل)ثايوريدو)بروبانوك اسد

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الخلاصة:

(3-(4-هيدروكسي فنل)-2-(3-(4-نايتروبنزويل) ثايوريدو بربانوك اسد) (HNP)هو ليكاند جديد تم تحضيره من تفاعل التايروسين مع(4-نايترو بنزويل ازوثايوسيانيت)، وقد تم تشخيص الليكاند بوساطة التحليل الدقيق للعناصر ،الأشعة تحت الحمراء والأشعة فوق البنفسجية- المرئية وطيف الرنين النووي المغناطيسي بعد تحضير الليكاند (HNP)حضرت سبع معقدات لأملاح بعض الفلزات ثنائية التكافؤ (المنغنيز، كوبلت، نيكل، نحاس،زنك، كادميوم و الزئبق) وشخصت باستعمال الأشعة تحت الحمراء والأشعة فوق البنفسجية-المرئية والتوصيلية المولارية و الحساسية المغناطيسية والامتصاص الذري. ومن خلال النتائج المستحصلة تم اعطاء المعقدات الصيغة العامة[M (HNP)2] اذ ان (M+2 = Mn, Co, Ni, Cu, Zn, Cd anHg). وقد اقترح الشكل رباعي السطوح لجميع المعقدات المحضرة عدا معقد النحاس ذو شكل مربع مستوي .

الكلمات المفتاحية : التايروسين، الفلزات ثنائية التكافؤ، 4- نايتروبنزويل ازوثايوسيانيت.