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(1H,13C-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\n\n= 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 (-NH2) 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](H2O)(ClO4) (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$_2$O)] 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 its 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**
- $^1$H and $^{13}$C–NMR were recorded using Ultra Shield 300MH$_2$, Switzerland at University of Al al-Bayt, Jordan.
- Melting point was recorded by using melting point meter KRUSS (A. KRUSS OPHTRONIC).
- FT-IR spectra were recorded as FT-IR using 3800 Shimadzu in the range of (4000-400) cm$^{-1}$ by using (KBr) disc.
- Electronic spectra were obtained using (UV-160 Shimadzu) spectrophotometer at 25$^\circ$C for 10$^{-3}$M solution DMSO.
- Molar Conductivity was measured at 25$^\circ$C for 10$^{-3}$M by using PhilipsPW.
- Digital micro elemental analysis (C.H.N.S) were performed using AcrloErba 1106 elemental 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-hydroxyphenyl)-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)$^\circ$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) MnCl₂.4H₂O 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 (CoCl₂.6H₂O, NiCl₂.6H₂O, CuCl₂.2H₂O, ZnCl₂, CdCl₂.2H₂O and HgCl₂) 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 'cm' mol⁻¹ In DMSO | μ_eff (B.M) |
|----------|----------------|---------|----------------|------------------------|------------------------------------|-------------|
| Ligand(HNP) C₁₇H₁₅O₆N₃S | 389.38 | Brown   | 158-160        | 6.61 (5.89)            | 14.5                               | 5.93        |
| [Mn(HNP)₂] | 831.69 | Light Green | 168-170      | 7.05 (6.91)           | 11.1                               | 4.91        |
| [Co(HNP)₂] | 835.68 | Green   | 166-168       | 7.03 (6.80)           | 15.1                               | 2.99        |
| [Ni(HNP)₂] | 835.44 | greenish yellow | 232-233 Dec. | 7.56 (7.01)           | 13.4                               | 1.78        |
| [Cu(HNP)₂] | 840.30 | Orang   | 220-222       | 7.76 (8.02)           | 15.8                               | 0.00        |
| [Zn(HNP)₂] | 842.13 | Yellow  | 230-231 Dec. | 12.64 (12.35)         | 14.0                               | 0.00        |
| [Cd(HNP)₂] | 889.16 | Yellow  | 200-202       | 20.52 (21.23)         | 11.8                               | 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 (1H-NMR) spectrum of the ligand (HNP) Figure (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: 1H-NMR spectrum of ligand (HNP)](image)

The (13C-NMR) spectrum of the ligand (HNP) Figure (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: 13C-NMR spectrum of ligand (HNP)](image)

The (FT-IR) spectrum of the free ligand (HNP), Figure (3), shows broad band at (2951, 3066) cm⁻¹ due to υ(OH, NH₂). While other absorption bands at (1701) cm⁻¹, (1327) cm⁻¹, (1600) cm⁻¹, (1246) cm⁻¹ due to υ(COOASY), υ(COOSYM), 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)](image)

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 π→π* [11]. The data of electronic spectrum of the free ligand (HNP) is registered in Table (3).
Baghdad Science Journal

Vol. 14(3) 2017

592

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 $\nu$(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 o(N-H) group [13] while the band at (1701) cm$^{-1}$ was assigned to the stretching of $\nu$(COO)$_{\text{sym}}$ 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 $\nu$(COO)$_{\text{sym}}$ 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 $\nu$(C=O)$_{\text{amid}}$ and $\nu$(C=S) either show no change or very little in their frequencies (1620-1604) cm$^{-1}$, (1230-1280) cm$^{-1}$ respectively; therefore, indicating do not coordinate to the metal ion. Metal-oxygen and metal-nitrogen were confirmed by the presence of the stretching tremor of $\nu$(M-N) and $\nu$(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 | v(O – H) | v(C=O) | v(COO) |
|-----------|----------|--------|--------|
| (HNP)     | 3606 cm⁻¹| 3174 cm⁻¹| 3170 cm⁻¹|
| [Mn(HNP)₂] | 3559 cm⁻¹| 1670 cm⁻¹| 1415 cm⁻¹|
| [Co(HNP)₂] | 3159 cm⁻¹| 1670 cm⁻¹| 1415 cm⁻¹|
| [Ni(HNP)₂] | 3174 cm⁻¹| 1666 cm⁻¹| 1411 cm⁻¹|
| [Cu(HNP)₂] | 3116 cm⁻¹| 1670 cm⁻¹| 1415 cm⁻¹|
| [Zn(HNP)₂] | 3217 cm⁻¹| 1689 cm⁻¹| 1435 cm⁻¹|
| [Cd(HNP)₂] | 3174 cm⁻¹| 1689 cm⁻¹| 1415 cm⁻¹|
| [Hg(HNP)₂] | 3174 cm⁻¹| 1689 cm⁻¹| 1415 cm⁻¹|

Electronic spectra for complexes and Magnetic moment

-[Mn(HNP)₂] \( d^8 \)

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 \( ^6A_1 \rightarrow ^4T_2 \) and \( ^6A_1 \rightarrow ^4T_1 \) respectively. The value of measured \( \mu_{\text{eff}} \) 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]](image)

- [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 \( ^4A_2 \rightarrow ^4T_1 \), \( ^4A_2 \rightarrow ^4T_1 \) and \( ^4A_2 \rightarrow ^4T_2 \) 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 \( \mu_{\text{eff}} \) 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 \( ^5T_1 (F) \rightarrow ^3T_1 (P)^3T_1 \rightarrow ^1A_1 \), and \( ^3T_1 \rightarrow ^3T_2 \), 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, which was equal to (0.751). These are the characteristics for Tetrahedral complexes of Ni\(^{2+}\) [19] The value of measured \( \mu_{\text{eff}} \) 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)\(^2\)B₁g \( \rightarrow ^2A_{1g} \), \( ^2B_{1g} \) transition respectively and the value of measured \( \mu_{\text{eff}} \) for Cu(II) showed \( \mu_{\text{eff}}(1.78) \) 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 ($\mu_{\text{eff}}$) 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            | $\lambda$(nm) | $\nu$(cm$^{-1}$) | ABC | $\varepsilon_{\text{max}}$ molar$^{-1}$cm$^{-1}$ | Transitions |
|----------------------|---------------|-----------------|-----|---------------------------------------------|-------------|
| Ligand (HNP)         | 272           | 36764           | 2.016 | 2016                                       | $\pi \rightarrow \pi^*$ |
| [Mn(HNP)$_2$]        | 277           | 36101           | 2.288 | 2228                                        | $^6A_1 \rightarrow ^4T_2$ |
|                      | 345           | 28985           | 1.248 | 1248                                        | $^6A_1 \rightarrow ^4T_2$ |
|                      | 813           | 12300           | 0.020 | 0                                             | L.F.         |
| [Co(HNP)$_2$]        | 278           | 35971           | 2.311 | 2311                                        | $^6A_1 \rightarrow ^4T_2$ |
|                      | 390           | 25641           | 0.990 | 990                                          | L.F.         |
|                      | 786           | 12722           | 0.020 | 20                                            | $^4A_2 \rightarrow ^4T_1$ |
|                      | 964           | 10373           | 0.018 | 18                                            | $^4A_2 \rightarrow ^4T_2$ |
| [Ni(HNP)$_2$]        | 275           | 36363           | 2.213 | 2213                                        | $^4T_1$ (F) $\rightarrow ^4T_1$ (P) |
|                      | 360           | 27777           | 0.755 | 755                                          | $^4T_1$ $\rightarrow ^4A_2$ |
|                      | 734           | 13623           | 0.025 | 25                                            | $^4T_1$ $\rightarrow ^4T_2$ |
|                      | 1011          | 9891            | 0.018 | 18                                            | C.T.         |
| [Cu(HNP)$_2$]        | 273           | 36630           | 2.056 | 2056                                        | $^2B_1g$ $\rightarrow ^2A_1g$ |
|                      | 813           | 12300           | 0.028 | 28                                            | $^2B_2g$ $\rightarrow ^2B_2g$ |
|                      | 865           | 11560           | 0.018 | 18                                            | L.F.         |
| [Zn(HNP)$_2$]        | 275           | 36363           | 2.114 | 2114                                        | L.F.         |
|                      | 538           | 18939           | 0.075 | 75                                            | C.T.         |
| [Cd(HNP)$_2$]        | 272           | 36764           | 1.957 | 1957                                        | L.F.         |
|                      | 345           | 28985           | 0.469 | 469                                          | C.T.         |
| [Hg(HNP)$_2$]        | 273           | 36630           | 2.069 | 2069                                        | L.F.         |
|                      | 352           | 28409           | 0.250 | 250                                          | 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 (FTIR, 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 bidentate 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).

![Chemical structure formula of the complexes](image-url)

Fig 7: The proposed chemical structure formula of the complexes
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تحضیر ودراسة طيفية لبعض معقدات الفلزات ثنائية التكافؤ مع 3-((4-هيدروكسي فنل)-2-(3-(4-نيتروبنزويل) ثايوريدو) بروبانوك اسد

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الخلاصة:
هو ليكاند جديد تم تحضيره (HNP) من تفاعل التايروسين مع (4-نابتيروبنزويل ازوثايوسيانيت)، وقد تم تشخيص الليكاند بوسطة التحليل الالقاحي للعناصر، الأشعة تحت الحمراء والأشعة فوق البنفسجية والأشعة تحت الحمراء والأشعة فوق البنفسجية. الليكاند (HNP) حضرت سبع معقدات لإنتاج بعض الفلزات ثنائية التكافؤ (المنغنيز، كوبالت، نيكلاج، نحاس، زئبق، كلاديميوم و الزئبق) وتشخصت باستعمال الأشعة تحت الحمراء والأشعة فوق البنفسجية. وتمت التوصيلية المولارية والحساسية المغناطيسية والامتصاص الذري، ومن خلال النتائج المستحصلة تم اعطاء المعقدات الصيغة العامة [M (HNP)]2 Mn، Co، Ni، Cu، Zn، Cd و Hg +2 = (M+2 = Mn، Co، Ni، Cu، Zn، Cd و Hg +2 = (M+2 = Mn، Co، Ni، Cu، Zn، Cd و Hg +2 = (M+2 = Mn، Co، Ni، Cu، Zn، Cd و Hg +2 = (M+2 = Mn، Co، Ni، Cu، Zn، Cd و Hg +2 = (M+2 = Mn، Co، Ni، Cu، Zn، Cd و Hg +2 = (M+2 = Mn، Co، Ni، Cu، Zn، Cd و Hg +2 = (M+2 = Mn، Co، Ni، Cu، Zn، Cd و Hg +2 = (M+2 = Mn، Co، Ni، Cu، Zn، Cd و Hg +2 = (M+2 = Mn، Co، Ni، Cu، Zn، Cd و Hg +2 = (M+2 = Mn، Co، Ni، Cu، Zn، Cd و Hg +2 = (M+2 = Mn، Co، Ni، Cu، Zn، Cd و Hg +2 = (M+2 = Mn، Co، Ni، Cu، Zn، Cd و Hg +2 = (M+2 = Mn، Co، Ni، Cu، Zn، Cd و Hg +2 = (M+2 = Mn، Co، Ni، Cu، Zn، Cd و Hg +2 = (M+2 = Mn، Co، Ni، Cu، Zn، Cd و Hg +2 = (M+2 = Mn، Co، Ni، Cu، Zn، Cd و Hg +2 = (M+2 = Mn، Co، Ni، Cu، Zn، Cd و Hg +2 = (M+2 = Mn، Co، Ni، Cu، Zn، Cd و Hg +2 = (M+2 = Mn، Co، Ni، Cu، Zn، Cd و Hg +2 = (M+2 = Mn، Co، Ni، Cu، Zn، Cd و Hg +2 = (M+2 = Mn، Co، Ni، Cu، Zn، Cd و Hg +2 = (M+2 = Mn، Co، Ni، Cu، Zn، Cd و Hg +2 = (M+2 = Mn، Co، Ni، Cu، Zn، Cd و Hg +2 = (M+2 = Mn، Co،Ni

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