

## Synthesis and Characterization of Some Metals Complexes of {N-[(Benzoyl Amino)-Thioxo Methyl] Proline}

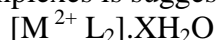
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### Abstract

A new ligand {N-[(benzoyl amino)-thioxo methyl] proline} was prepared by the reaction of benzoyl isothiocyanate with proline. The ligand was characterized by FTIR, UV-Visible, and <sup>1</sup>HNMR. Some complexes of the ligand were synthesized and characterized on the basis of their FTIR, UV-Visible spectroscopy, conductive measurements and magnetic susceptibility.

From the results obtained, the following general formula has suggested for the prepared complexes is suggested



Where: M = Mn<sup>+2</sup>, Fe<sup>+2</sup>, Co<sup>+2</sup>, Ni<sup>+2</sup>, Cu<sup>+2</sup>, Zn<sup>+2</sup>, Cd<sup>+2</sup> and Hg<sup>+2</sup>

X = 3, 0, 2, 0, 4, 0, 0 and 0

L<sup>-</sup> = {N-[(benzoyl amino)-thioxo methyl] prolinato}

Keywords: Proline, Benzoyl isothiocyanate, Amino acids, Transition metals.

### Introduction

Amino acids complexes have considerable biological activity, such as antitumor properties and mixed ligand complexes of Zn(II) and Cd(II) containing ceftriaxone antibiotic and amino acids have been prepared and characterized by elemental analysis, spectral, biological and thermal studies. Complexes of thiourea derivatives of amino acids with transition metals [1-6] and rare earth metals have been prepared [7-9], also Pd(II)-N-heterocyclic carbene complexes derived from proline have been successfully synthesized in good yields and their structures have been characterized by X-ray. The tolyl porphyrin and dithiocarbamate proline derivatives and their iron, cobalt and tin complexes have been synthesized and characterized by IR, UV-Vis, MS and spectroscopy studies also a solid Co<sup>II</sup> complex with L-proline as ligand was synthesized and characterized using spectroscopic studies, magnetic data, UV-Vis spectroscopy. Complexes of transition metals (Zn, Cd, Ni, Co, Mn) with 1-(3-mercapto-2-D-methyl-1-oxopropyl)-1-proline has been studied and discussed (10-14).

The aim of this work is preparation of some transition metals complexes of {N-[(benzoyl amino)-thioxo methyl] proline}.

### Experimental

#### *Materials and Measurements:*

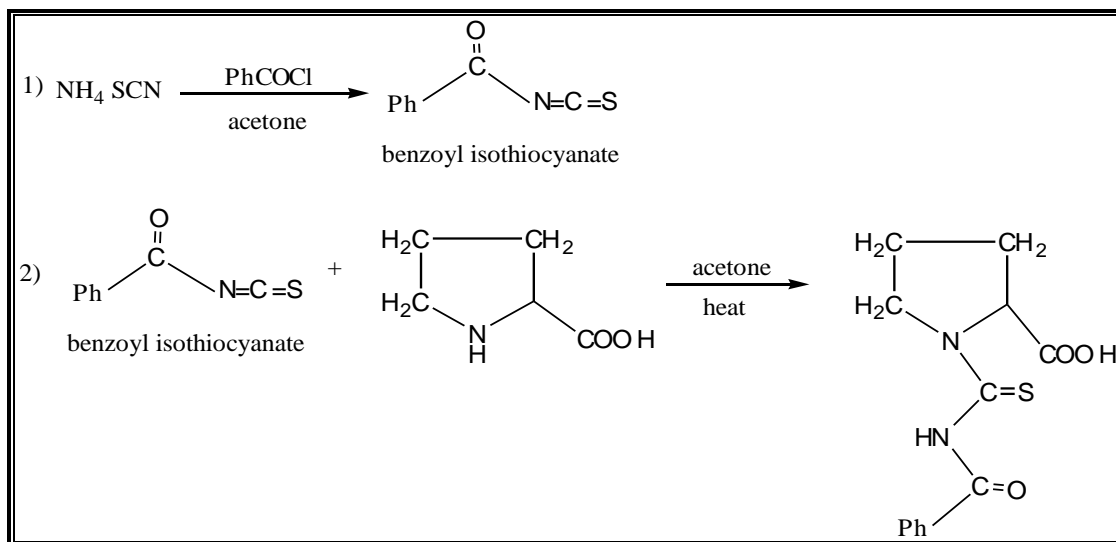
Metal salts (MnCl<sub>2</sub>.4H<sub>2</sub>O, FeSO<sub>4</sub>.7H<sub>2</sub>O, CoCl<sub>2</sub>.6H<sub>2</sub>O, NiCl<sub>2</sub>.6H<sub>2</sub>O, CuCl<sub>2</sub>.2H<sub>2</sub>O, ZnCl<sub>2</sub>, CdCl<sub>2</sub>.H<sub>2</sub>O and HgCl<sub>2</sub>) were obtained from Fluka, Merck in high purity. Proline, benzoyl chloride and ammonium thiocyanate (Fluka). (<sup>1</sup>HNMR) were recorded using ultra shield 300 MHz, Switzerland, at University of Al al-Bayt, Jordan. Conductivity measurements were carried out using Philips PW. Digital - meter. The FTIR spectra in the region (4000-200) cm<sup>-1</sup> were recorded using (Shimadzu, FTIR-8300). Infrared spectra photometer as cesium-iodide disc. The UV-Vis spectra were recorded using (Shimadzu UV-Vis 160A), UV-Vis spectrophotometer in dimethylformamide solution (10<sup>-3</sup>M). Metal contents of the complexes were determined by Atomic absorption using (Shimadzu AA680G) Atomic absorption spectrophotometer. The magnetic moments (μ<sub>eff</sub> BM) were calculated on Faraday method using (Balance Magnetic susceptibility model MSB-MKT). Melting points were determined by using (Stuart-melting point apparatus).

**Synthesis of the Ligand:****(1) Preparation of the benzoyl isothiocyanate [15]**

A mixture of benzoyl chloride (3.05ml, 1mmol) and ammonium thiocyanate (2gm, 1mmol) in 25 ml acetone was refluxed with stirring for 1 hour, and then filtered; the filtrate was used for further reaction.

**2) Preparation of {N-[(benzoyl amino)-thioxo methyl] proline} (L)**

(3.02 gm, 1mmol) of proline in 20 ml acetone were rapidly added to the above solution to maintain vigorous reflux. After refluxing for 6 hours, the resulting solid was collected, washed with acetone and recrystallized from ethanol (m.p. = 104 °C).



**Scheme (1) Preparation {N-[(benzoyl amino)-thioxo methyl] proline} (L).**

**Synthesis of the Complexes:**

(0.25 gm, 0.9 mmol) of the ligand was dissolved in 25ml of methanol containing (0.05 gm, 0.9 mmol) of KOH. A solution of metal salts (0.06-0.1 gm, 0.44-0.4 mmol) in ethanol was added drop wise to the mixture, and the precipitate formed immediately.

After stirring the mixture at room temperature for 2 hours, the precipitate was collected by filtration, washed with ethanol and dried.

**Results and Discussion**

The isolated complexes were crystalline solids, soluble in some of the common solvents such as dimethylformamide, dimethylsulphoxide, they are relatively thermally stable. The conductivity measurements in DMF indicated the non-electrolyte behavior; Table (1) includes the physical properties.

The analytical data confirmed the (1:2) (M:L) where  $M = [\text{Mn}^{+2}, \text{Fe}^{+2}, \text{Co}^{+2}, \text{Ni}^{+2}, \text{Cu}^{+2}, \text{Zn}^{+2}, \text{Cd}^{+2} \text{ and } \text{Hg}^{+2}]$  composition of the complexes. The magnetic measurements ( $\mu_{\text{eff}}$  BM) for the complexes are listed in Table (1) also.

**Spectral Studies:****<sup>1</sup>HNMR spectrum for the ligand (L):**

The <sup>1</sup>HNMR spectrum of (L), Fig.(1) showed the following signals: pentet at  $\delta(1.80-2.30)$  ppm for (2H, CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>2</sub>), singlet at  $\delta(2.50)$  ppm and  $\delta(4.02)$  ppm for solvent (DMSO), quartet at  $\delta(3.09-3.12)$  ppm for (2H,CH<sub>2</sub>-CH), triplet at  $\delta(3.67-3.82)$  ppm for (2H,CH<sub>2</sub>-N), singlet at  $\delta(4.47-4.48)$  ppm for (1H, NH), triplet at  $\delta(4.67-4.70)$  ppm for (1H,CH-COOH), multiplet at  $\delta(7.37-7.94)$  ppm for (5H, aromatic) and broad peaks at  $\delta(10.9)$  ppm for (1H, COOH).

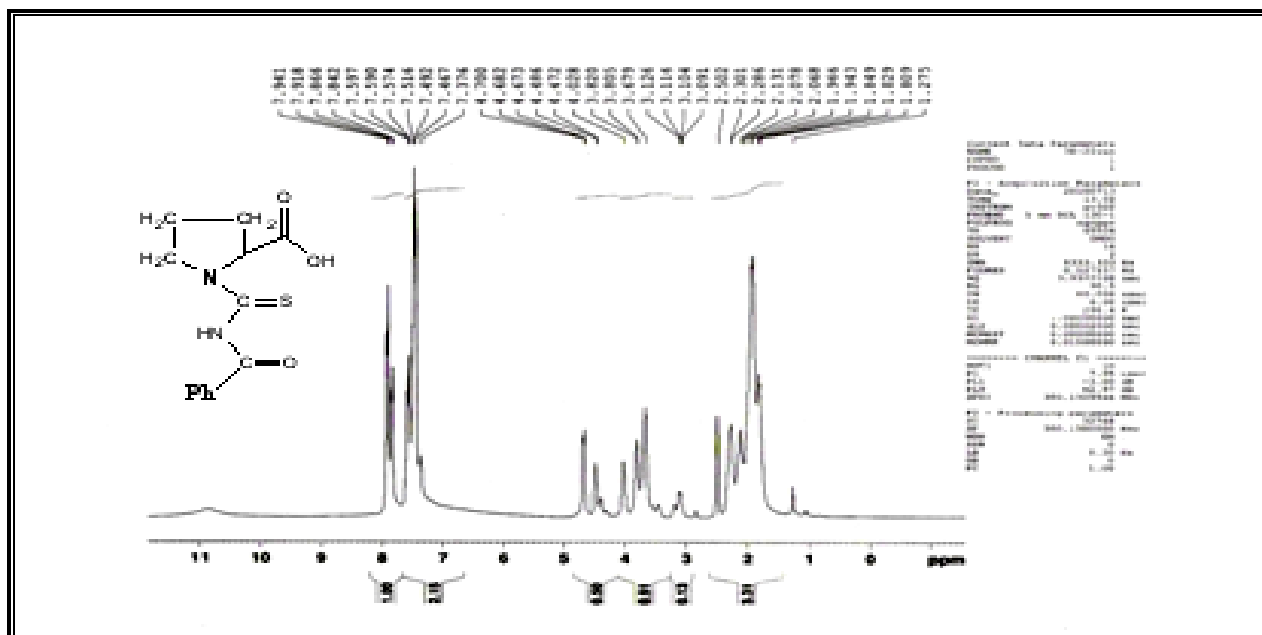


Fig. (1)  $^1\text{H}$ NMR spectrum for the ligand (N-[(benzoyl amino)-thioxo methyl] proline) (L).

### Infrared Spectra:

In order to get further information about the coordination behavior of the ligand {N-[(benzoyl amino)-thioxo methyl] proline} with metal ions, comparisons of the infrared spectra of the free ligand, Fig.(Y) and its complexes, Fig. (Z) were made. The important absorption at assignment are listed in Table(2). The band at  $(3356)\text{ cm}^{-1}$  in the spectrum of the free ligand assigned to the  $\nu(\text{N-H})$ , while another absorption band appeared at  $(1732)\text{ cm}^{-1}$  could be explained as  $\nu(\text{COO})_{\text{asym}}$  where the  $\nu(\text{OCO})_{\text{sym}}$  was noticed at  $(1427)\text{ cm}^{-1}$  [16-21], in addition to absorption band at  $(1211)\text{ cm}^{-1}$  which assigned to  $\nu(\text{C=S})$ [22].

### The FTIR spectra of complexes:

These spectra exhibited a marked difference between bands belonging to the stretching vibration of  $\nu(\text{N-H})$  of the amine group in the range between  $(3448-3383)\text{ cm}^{-1}$  shifted to higher frequencies by  $(92-27)\text{ cm}^{-1}$  suggesting the possibility of the coordination of ligand through the nitrogen atom at the amine group [23-25] absorption assigned for  $\nu(\text{OCO})_{\text{sym}}$  was noticed at the range  $(1373-1126)\text{ cm}^{-1}$  shifted to a lower position by  $(301-54)\text{ cm}^{-1}$ , while the band caused by  $\nu(\text{OCO})_{\text{asym}}$  appeared between  $(1616-1512)\text{ cm}^{-1}$  shifted to lower frequencies by  $(220-116)\text{ cm}^{-1}$  which indicates the

coordination of the carboxylic group to the central metal ion [24,25].

The stretching vibration band  $\nu(\text{C=S})$  was found in the range  $(1087-1026)\text{ cm}^{-1}$  shifted to a lower frequency by  $(185-124)\text{ cm}^{-1}$  which means that the sulphur atom was involved in the coordination [26, 27].

Metal-nitrogen and metal-oxygen bands were confirmed by the presence of the stretching vibration of  $\nu(\text{M-O})$ ,  $\nu(\text{M-N})$  and  $(\text{M-S})$  around  $(470-447)\text{ cm}^{-1}$ ,  $(617-528)\text{ cm}^{-1}$ , and  $(439-416)\text{ cm}^{-1}$  respectively.

### Electronic spectra:

The absorptions and assignments related to the ligand and its complexes are listed in Table (3). The ligand (Fig.(4)) exhibited an absorption band in (UV) region at wave number  $(46296)\text{ cm}^{-1}$ , which may be attributed to  $(\pi \rightarrow \pi^*)$  transition, other band of low intensity appeared at  $(32051)\text{ cm}^{-1}$  was expressed at the  $(n \rightarrow \pi^*)$  [28].

### The spectra of complexes

-[Mn L<sub>2</sub>].3H<sub>2</sub>O ( $d^5$ )

The yellow complex spectrum showed two absorptions at  $(46511)\text{ cm}^{-1}$  and  $(31746)\text{ cm}^{-1}$  attributed to  $(\text{C.T})$  and  $6A_{1g} \rightarrow 4T_{1g(G)}$  transition respectively [29,30].

-[Fe L<sub>2</sub>] ( $d^6$ )

In the spectrum, the orange complex showed two absorptions at  $(32894)\text{ cm}^{-1}$  and

(13661)  $\text{cm}^{-1}$  attributed to (C.T) and  $5T_{2g} \rightarrow 5E_g$ , respectively [31].

-[Co L<sub>2</sub>].2H<sub>2</sub>O (d<sup>7</sup>)

The spectrum of the green, (Fig.(5)) exhibited the following bands at (46296)  $\text{cm}^{-1}$ , (31847)  $\text{cm}^{-1}$ , (16474)  $\text{cm}^{-1}$ , (13568)  $\text{cm}^{-1}$  which have been assigned as (C.T),  $4T_{1g}(f) \rightarrow 4T_{1g}(p)$ ,  $4T_{1g} \rightarrow 4A_{2g}$  and  $4T_{1g} \rightarrow 4T_{2g}$  respectively. The Racah inter electronic repulsion parameter  $\tilde{B}$  found to be (507.8)  $\text{cm}^{-1}$ , the ratio  $\beta = \tilde{B} / B^0$  comes out to be (0.523), these parameters are accepted for cobalt (II) octahedral complexes [32, 33].

-[Ni L<sub>2</sub>] (d<sup>8</sup>)

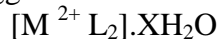
The spectrum of pink complex exhibited the following absorptions at (46296)  $\text{cm}^{-1}$ , (32894)  $\text{cm}^{-1}$ , (18181)  $\text{cm}^{-1}$  and (14265)  $\text{cm}^{-1}$  these bands are characteristic of octahedral nickel(II) [34] complex and were assigned to the transition (C.T),  $3A_{2g} \rightarrow 3T_{1g}(p)$ ,  $3A_{2g} \rightarrow 3T_{1g}$  and  $3A_{2g} \rightarrow 3T_{2g}(f)$  respectively, and the  $\tilde{B}$  found to be (552)  $\text{cm}^{-1}$  and  $\beta = \tilde{B} / B^0$  comes out to be (0.530).

-[Cu L<sub>2</sub>].4H<sub>2</sub>O (d<sup>9</sup>)

The deep green complex of Cu (II) gave two bands at (33112)  $\text{cm}^{-1}$  and (15625)  $\text{cm}^{-1}$  caused by (C.T) and  $2E_g \rightarrow 2T_{2g}$  transitions respectively [35,36].

-The white complexes [ZnL<sub>2</sub>], [CdL<sub>2</sub>] and [HgL<sub>2</sub>] where the electronic configuration of the metals is (d<sup>10</sup>) which confirms the absence of any (d→d) transitions [34].

According to spectral data as well as those obtained from elemental analyses, the chemical structure of the complexes may be suggested as:

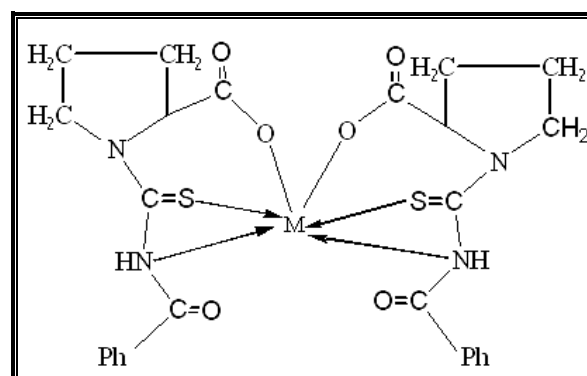


Where:

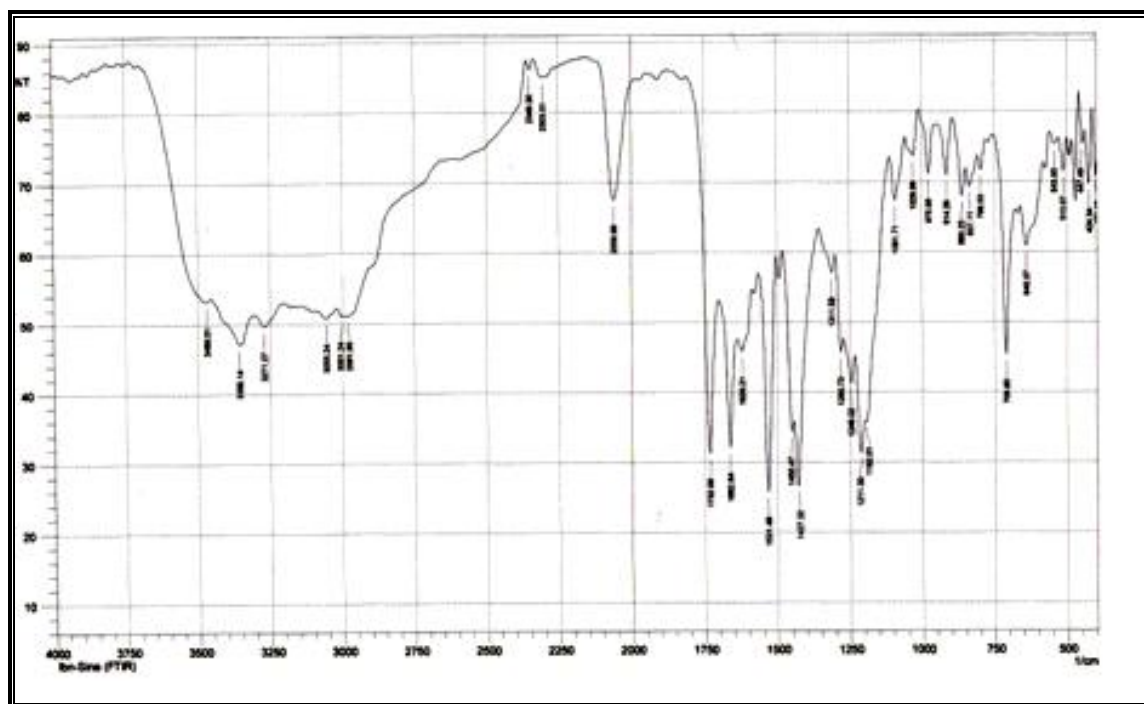
M = Mn<sup>+2</sup>, Fe<sup>+2</sup>, Co<sup>+2</sup>, Ni<sup>+2</sup>, Cu<sup>+2</sup>, Zn<sup>+2</sup>, Cd<sup>+2</sup>, Hg<sup>+2</sup>

X = 3, 0, 2, 0, 4, 0, 0, 0

L<sup>-</sup> = {N-[(benzoyl amino)-thioxo methyl] prolinato}



*General structure of complexes.*



*Fig. (2) FTIR spectrum of Ligand (N-[(benzoyl amino)-thioxo methyl] proline) (L).*

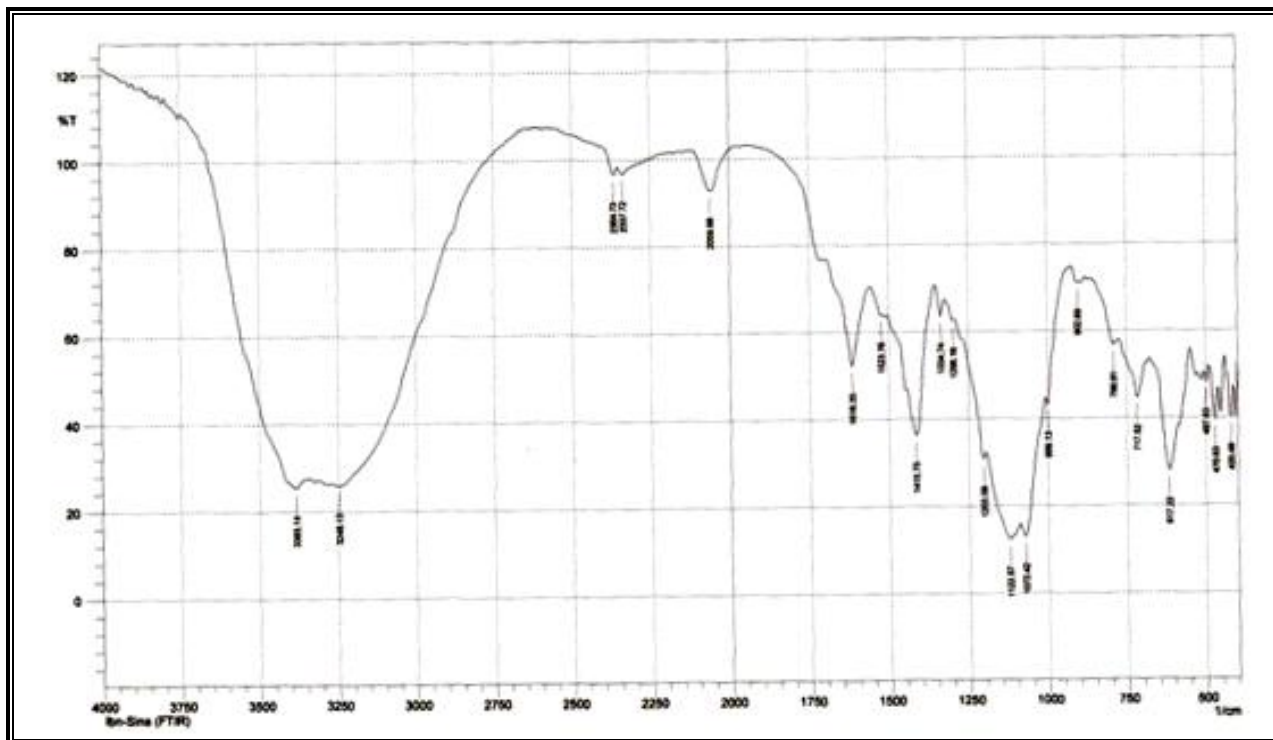


Fig. (3) FTIR spectrum of complex [Fe(L)<sub>2</sub>].

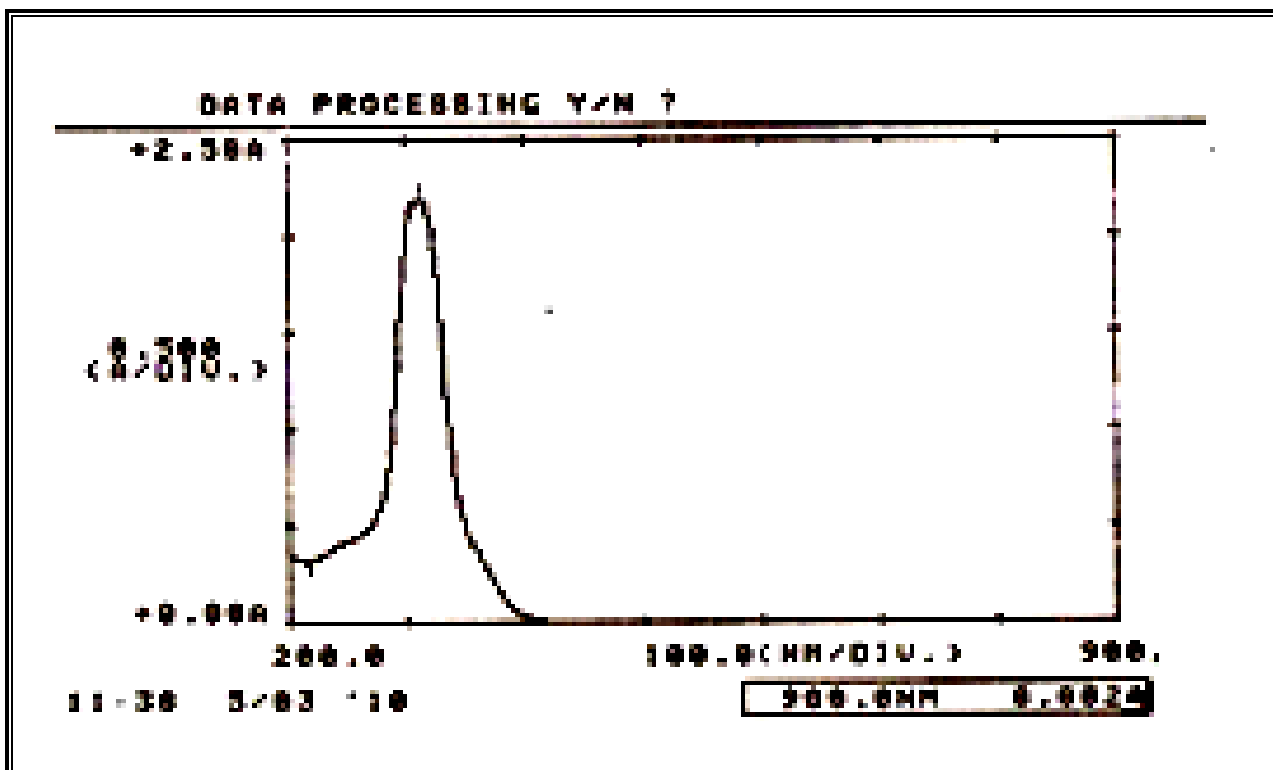


Fig. (4) UV-Vis Spectrum of Ligand (N-[(benzoyl amino)-thioxo methyl] proline) (L).

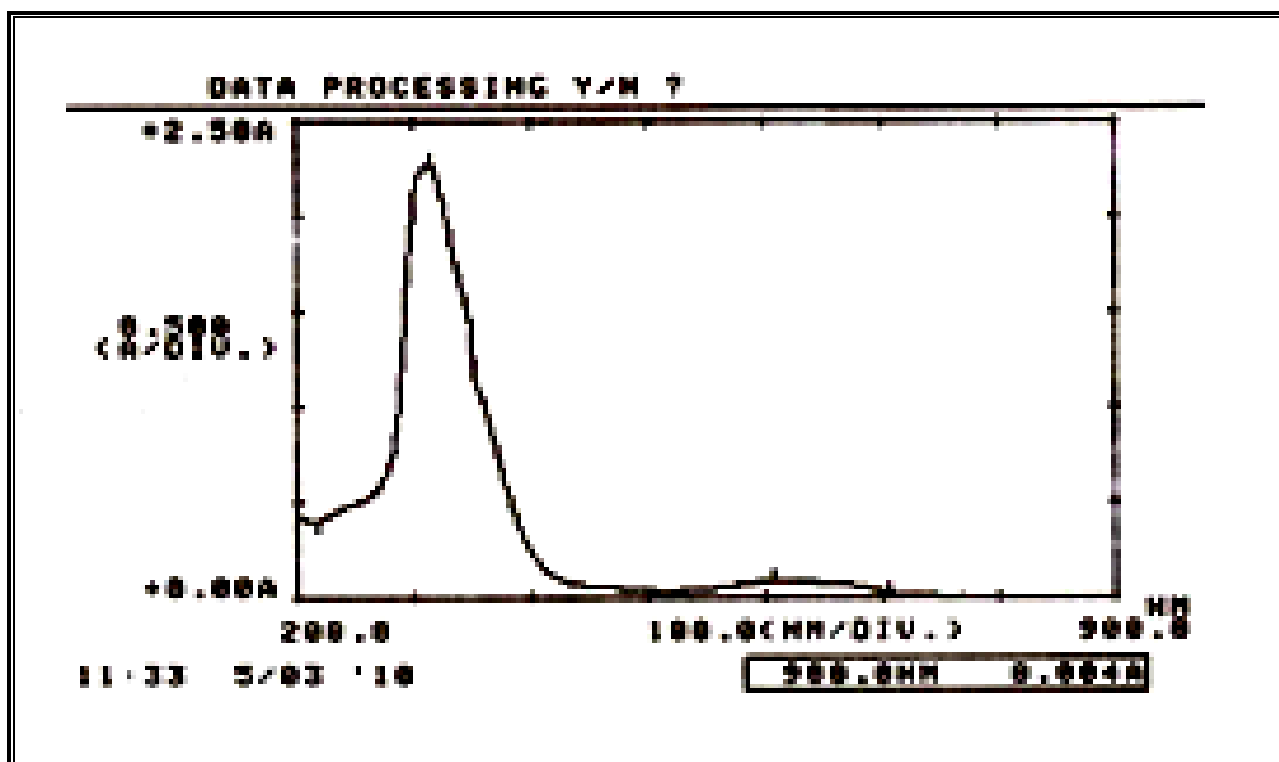


Fig. (5) UV-VIS Spectrum of complex  $[Co(L)_2].2H_2O$ .

Table (1)  
Physical properties for the free ligand and its complexes.

Complexes	Color	Dec. C°	M% Calculate (Found)	Molar conductivity ( $ohm^{-1}.cm^2.mol^{-1}$ ) in absolute DMF $10^{-3}M$	$\mu_{eff}(BM)$
Ligand (N-[(benzoylamino)- thioxo methyl] proline)	Yellow	104° M.P	–	8.50	–
$[Mn L_2]. 3H_2O$	Yellow	185 M.P	8.26 (8.21)	20.2	0.43
$[Fe L_2]$	orange	270 D	9.13 (9.44)	6.82	5.20
$[Co L_2]. 2H_2O$	green	280 D	9.05 (9.00)	29.3	4.62
$[Ni L_2]$	pink	210 D	9.55 (10.50)	30.6	2.11
$[Cu L_2].4H_2O$	Deep green	180 M.P	9.19 (9.29)	16.75	1.72
$[Zn L_2]$	white	225 M.P	10.52 (10.59)	23.1	0.00
$[Cd L_2]$	white	210 M.P	16.82 (17.33)	31.8	0.00
$[Hg L_2]$	white	260 D	26.51 (26.20)	32.2	0.00

**Table (2)**  
**The characteristic infrared of ligand {N-[(benzoyl amino)-thioxo methyl] proline} and its complexes.**

Complexes	$\nu(N-H)$	$\nu(C=O)$	$\nu(OCO)_{asym}$	$\nu(OCO)_{sym}$	$\nu(C=S)$	$\nu(M-S)$	$\nu(M-O)$	$\nu(M-N)$
Ligand	3356	1662	1732	1427	1211 798	–	–	–
[Mn L <sub>2</sub> ]. 3H <sub>2</sub> O	3441	1620	1531	1373	1026 713	428	466	547
[Fe L <sub>2</sub> ]	3383	1616	1523	1334	1072 790	420	470	617
[Co L <sub>2</sub> ]. 2H <sub>2</sub> O	3410	1589	1512	1334	1026 713	439	459	547
[Ni L <sub>2</sub> ]	3410	1689	1585	1338	1087 713	418	451	528
[Cu L <sub>2</sub> ]. 4H <sub>2</sub> O	3429	1627	1512	1334	1026 709	424	459	543
[Zn L <sub>2</sub> ]	3448	1631	1604	1334	1072 709	420	447	528
[Cd L <sub>2</sub> ]	3433	1685	1593	1334	1072 713	416	447	559
[Hg L <sub>2</sub> ]	3421	1639	1616	1126	1037 779	416	455	543

**Table (3)**  
**UV-Visible absorptions for the free ligand {N-[(benzoyl amino)-thioxo methyl] proline} and its complexes in DMF as a solvent in (10<sup>-3</sup> M).**

Complexes	$\lambda_{max}(nm)$	Wave number $cm^{-1}$	$\epsilon_{max}L.M^{-1}.cm^{-1}$	Assignment
Ligand	216 312	46296 32051	315 2195	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$
[Mn L <sub>2</sub> ].3H <sub>2</sub> O	215 315	46511 31746	344 2323	C.T. ${}^6A_{1g} \rightarrow {}^4T_{1g(G)}$
[Fe L <sub>2</sub> ]	304 732	32894 13661	617 30	C.T. ${}^5T_{2g} \rightarrow {}^5E_g$
[Co L <sub>2</sub> ].2H <sub>2</sub> O	216 314 607 737	46296 31847 16474 13568	398 2278 86 7	C.T. ${}^4T_{1g} \rightarrow {}^4T_{1g(P)}$ ${}^4T_{1g} \rightarrow {}^4A_{2g}$ ${}^4T_{1g} \rightarrow {}^4T_{2g}$
[Ni L <sub>2</sub> ]	216 304 550 701	46296 32894 18181 14265	302 1735 475 3	C.T. ${}^3A_{2g} \rightarrow {}^3T_{1g(P)}$ ${}^3A_{2g} \rightarrow {}^3T_{1g}$ ${}^3A_{2g} \rightarrow {}^3T_{2g(F)}$
[Cu L <sub>2</sub> ].4H <sub>2</sub> O	302 640	33112 15625	1470 80	C.T. ${}^2E_g \rightarrow {}^2T_{2g}$
[Zn L <sub>2</sub> ]	216	46296	331	C.T.
[Cd L <sub>2</sub> ]	315	31746	2445	C.T.
[Hg L <sub>2</sub> ]	348	28735	1091	C.T.

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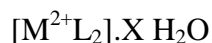


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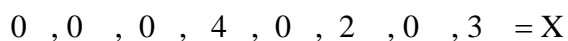
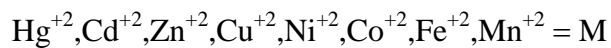
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### الخلاصة

يتضمن هذا البحث تحضير الليكاند الجديد {N-} (بنزويل امينو)- ثايوكسومثيل [برولين] بوساطة تفاعل بنزويل ايزو ثايوسيانات مع برولين وقد شخص هذا الليكاند مع معقداته المحضرة بالطرائق الطيفية ومنها (طيف الرنين النووي المغناطيسي  $^1\text{HNMR}$  والاشعة تحت الحمراء والاشعة المرئية - فوق البنفسجية) والتوصيلية المولارية والحساسية المغناطيسية، ومن نتائج هذه الدراسات التشخيصية امكن اعطاء الصيغة المقترحة العامة لهذه المعقدات وكالاتي:



حيث:



{بروليناتو}