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Synthesis, structural and Anti-Bacterial Study of Mn(II), Co(II), Zn(II), and Hg(II) Complexes with New Ligand 4-nitro-N-(pyrimidin-2-ylcarbamothioyl) benzamide

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Abstract

Using a 2-Amino pyrimidine and 4-nitrobenzoyl is othiocyanate, a novel ligand 4-nitro-N - (pyrimidin-2-ylcarbamothioyl) benzamide (L') has been created. Four of the ligand's transition metal complexes have been synthesized from Mn(II),Co(II),Zn(II) and Hg(II). The ligand and its complexes were characterized by using the UV-vis spectra, FT-IR, C.H.N.S., and ^{1} H, ^{13} CNMR for ligand while magnetic susceptibilities, conductivity measurements, and atomic absorptions for complexes. The behavior of the ligand is comparable to that of a bidentate coordinating through atoms S and O. According to the data, the chemical formula of every complex created was $[M(L)_2Cl_2]$ (M (II) = Mn, Co, Zn and Hg) . An octahedral geometrical structure was proposed for each complex. The metal and ligand complexes exhibited good antibacterial activity against Escherichia coli and Staphylococcus aureus.

Introduction

From both a theoretical and practical standpoint, Schiff base are compounds derived from pyrazine have been primarily prepared as classes of organic reagents [1]. Azomethine group is (-CH=N-) that results from the condensation of ketones or aldehydes with aromatic amines and has been used to describe Schiff base complexes [2-4]. Because the azomethine group is the characteristic functional group of Schiff bases in aromatic compounds, these reagents act as chromogenic in detecting trace amounts of transition metals, including Ni, in particular, natural food samples [5].

Additionally, ligands with at least two donor atoms concentrated in the coordination chemistry have been shown to tend to coordinate with different metal ions; coordination compounds are defined as specific metal ions encircled by ligands [6]. Ligands are molecules or ions that donate electrons to metal ions, forming coordinate bonds [7-9].

Because of their potential action in transition metal complexes, bioligands are extremely important. In several fields, including pharmacology, the Schiff bases are crucial because they have anti-bacterial, anti-fungal, and anti-cancer properties [10-12]. This study reports the synthesis and characterization of a new Schiff base ligand and its Mn(II), Co(II), Zn(II), and Hg (II) complexes.

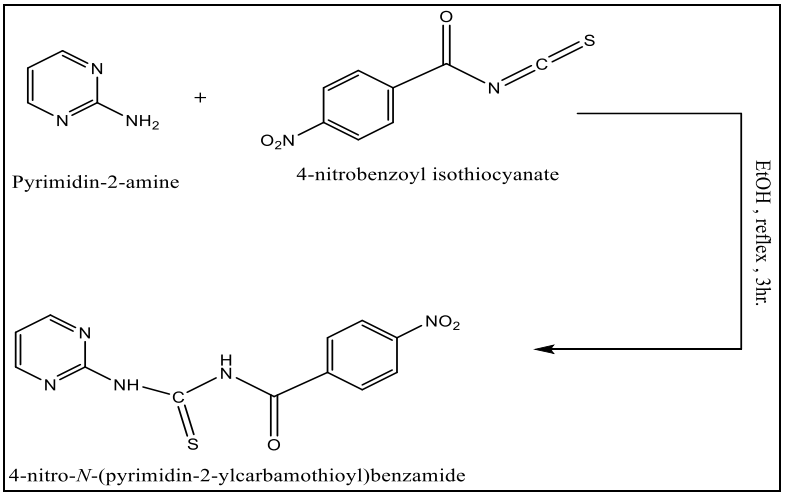
Materials and Methods

Physical Measurements and Materials

4-nitrobenzoyl isothiocyanate and pyridine-2-amine were obtained from FLUKA company 99%, Ethanol from BDH 98%, and the metal chloride from Merck company with the highest quality. Applying elemental analysis through the Ibn Sina company's (EURO EA-3000 Single) micro-analytical instrument for C.H.N.S. element analysis. By using a Shimadzu UV-Vis1700 spectro-photometer, the absorption spectra of a solution containing complexes in DMSO at 25 °C were recorded. FTIR spectra were registered at a range of ($4000cm^{-1}$ -$400cm^{-1}$) and ($4000cm^{-1}$ -$200cm^{-1}$) ranges of the ligand and metal complexes on Shimadzu IR470 spectra utilizing the CsI and KBr pellets. Molar conductivity measurements were performed on 10^{-3} M solutions of the complexes in ethanol at room temperature by using a PW-9526 digital conductivity meter. DMSOd-6 has been used to record ^{1}H,^{13}CNMR spectra at the Bruker 400 MHz spectrometer. Using a potentiometric titration procedure on a 686-Titro Processor665 Dosim A-Metrohm/Swiss, the chloride content is specified. Additionally, Stuart used an electrothermal device to determine the melting points; the Shimadzu (AA) 680 G atomic absorption spectrophotometer was used to specify the metals and melting points.

Ligand preparation

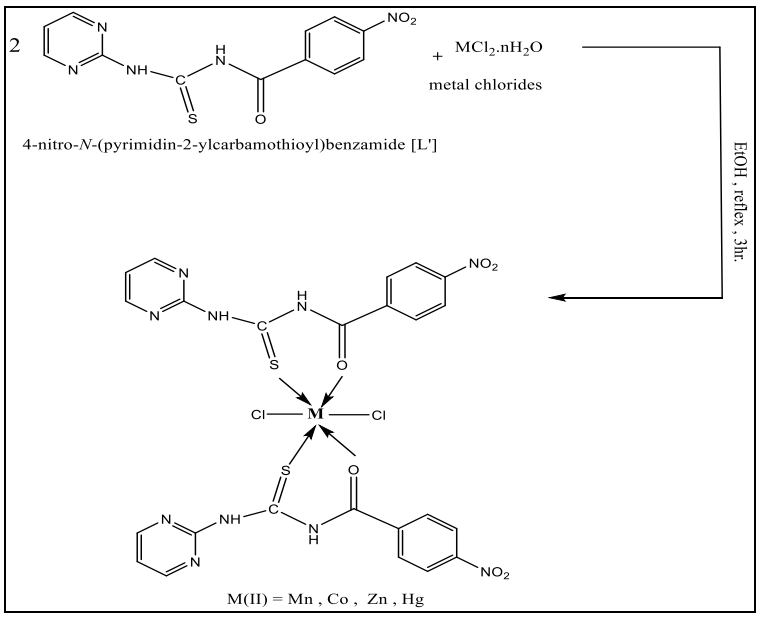
4-nitrobenzoyl isothiocyanate (0.30g, 0.0014 mol) and pyridine-2-amine (0.13 g, 0.0014 mol) in 15 mL ethanol were refluxed for three hours. dark brown crystals of 4-nitro-N-(pyrimidin-2-ylcarbamothioyl) benzamide were obtained by cooling, filtering, and recrystallizing the reaction mixture; the yield was 78 %, with m.p. = 199–201 ^{o}C (Scheme 1) [13].



Scheme 1: Ligand [L'] Preparation.

Metal complexes preparation

The metal complexes were prepared by reacting the ligand [L'] (0.425 g, 0.004 mol) with the corresponding metal chlorides in a 1:2 molar ratio. [L'] was dissolved in 20mL of absolute ethanol. A separate solution of the appropriate metal chloride ($MnCl_2 \cdot 4H_2O$ 0.29 g , $CoCl_2 \cdot 6H_2O$ 0.35g , $ZnCl_2$ 0.20 g , or $HgCl_2$ 0.40 g) (0.0015 mol) in 20 mL of absolute ethanol was prepared. The hot metal chloride solution was added dropwise to the ligand solution then the mixture was refluxed for 3 hours at 72 ^{0}C. Filtered the colored precipitate and washed it with ethanol. The product was dried under vacuum at room temperature to afford the desired metal complex. The physical properties and analytical data of the synthesized complexes are presented in Table 1. (Scheme 2)



Scheme 2: Preparation of the Ligand's [L'] complexes.

Table (1) physical properties of [L'] and their complexes.

No	Empirical formula	Color	M.Wt g/mol	MP °C	Elemental microanalysis, (Calc. %)						
					C	H	N	O	S	Metal	Cl
1	$C_{12}H_9N_5O_3S$	Dark Brown	303	199-201	47.44 (47.52)	2.72 (2.99)	22.97 (23.09)	15.75 (15.83)	10.44 (10.57)	-	-
2	$[Mn(L')_2Cl_2]$	Deep Yellow	732	222-225	39.22 (39.36)	2.36 (2.48)	19.01 (19.12)	12.99 (13.11)	(8.58) (8.75)	(7.38) (7.50)	9.55 (9.68)
3	$[Co(L')_2Cl_2]$	Deep Blue	736	237-239	39.101 (39.14)	2.33 (2.46)	18. 90 (19. 02)	12.91 (13.04)	(8.59) (8.71)	7.88 (8.00)	9.51 (9.63)
4	$[Zn(L')_2Cl_2]$	Yellow	742	229-231	38.69 (38.80)	2.32 (2.44)	18.75 (18.86)	12.79 (12.92)	(8.55) (8.63)	7.90 (8.09)	9.33 (9.45)
5	$[Hg(L')_2Cl_2]$	Yellow	878	224-226	32.75 (32.83)	1.85 (2.07)	15.78 (15.95)	10.77 (10.93)	2.22 (2.31)	22.37 (22.48)	7.82 (8.07)

Results and Discussion

Ligand (L')

The bands at (1662cm^{-1}) and (1168 cm^{-1}) in the ligand (L') FTIR spectra were assigned to υ (C=O amide) and υ (C=S) , while a distinct band (3329cm^{-1}) that corresponds to υ N-H [14,15] . The bands (C=O amide) and υ (C=S) have appeared in the spectrum of ligand with the new band N-H that mean the ligand was formed. (Figure 1).

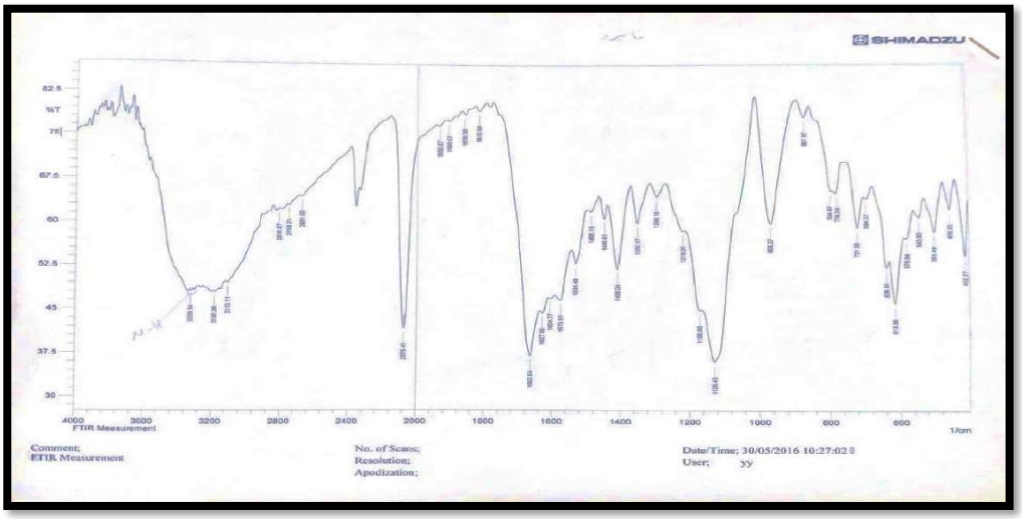


Fig. 1: The spectrum of FT-IR for [L'].

In the DMSO-d^6 solvent, the free ligand (L') ^{1}H-NMR spectrum (Figure 2) revealed several signals, including a singlet peak for the DMSO at δ (2.5) ppm, multiplet peaks for the aromatic protons at δ (6.73 - δ8.94) ppm, a singlet peak for (1H, NH sec. amide) at δ (9.80) ppm, and a singlet peak for (1H, NH -CS) at δ (11.60) ppm [16,17].

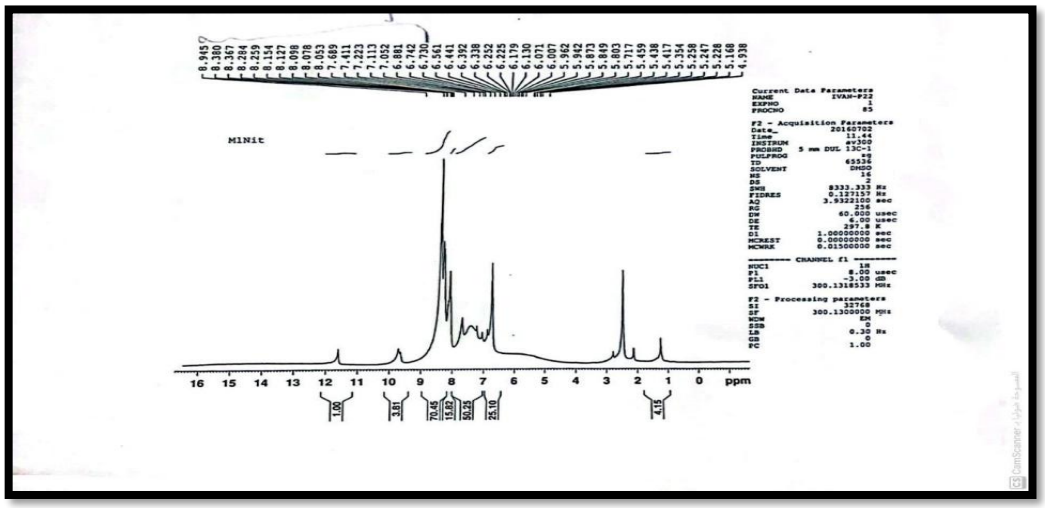


Fig. 2: The spectrum of ^{1}HNMR for [L'].

The ^{13}C NMR spectrum of [L'] in DMSO-d6 are shown in Figure (3). These spectra show several signals, including δ (38.60-40.02 ppm) for the DMSO, δ (109.91 and 158.08 ppm) for the aromatic carbons , while δ (159.82 ppm) for the C=O sec. amide, and δ (166.15 ppm) for (C=S) [18, 19].

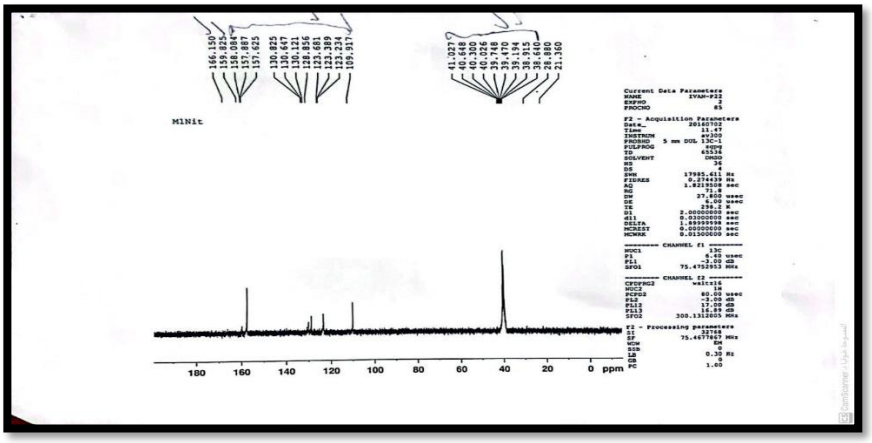


Fig. 3: ^{13}CNMR spectrum of [L'].

The UV-Vis spectrum of the unbound ligand [L'] a high-intensity absorption peak at (300 nm) (33333 cm^{-1}) is shown in Fig.(4.) This absorption is attributed to an electronic transition of the π → π* type [20,21]. Table 3 presents the electronic spectrum data for the free ligand [L'].

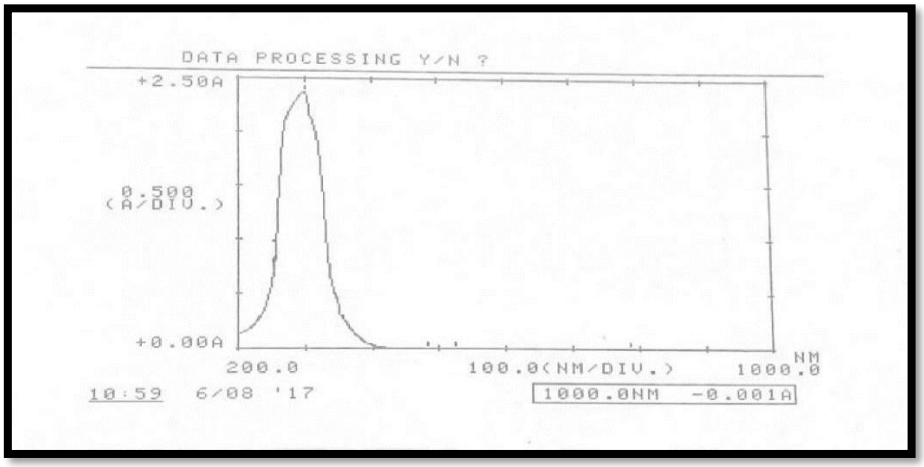


Figure 4: The spectrum of UV-Vis [L'].

The Complexes

FTIR Spectra

The stretching vibration band υ(N-H) indicated that it does not coordinate with metal ions since it either showed no changes or minimal frequency values (3323-3332) cm^{-1}. Coordination

between an Oxygen atom in the (C=O) group and a Sulfur atom in the (C=S) group with metal ions occurred when the stretching frequency of the ligand's (C=O) moved to the higher frequency at rang (1675-1681) cm^{-1} , it was shifted by (13- 19) degree · [22-24] , and the stretching frequency of the ligand's (C=S) group moved to the higher frequency at rang (1184-1213) cm^{-1} , it was shifted by (16- 45) degree . New bands absent in the ligand spectrum in the ligand spectra, υ (M-O) at rang (462- 474) cm^{-1}, υ (M-S) at rang (416-430) cm^{-1} and υ (M-Cl) at rang (243- 258) cm^{-1} [25-27]. The FT-IR spectra for Mn and Hg complexes are displayed in Fig (5) , Fig.(6) . Table (2) lists the produced compounds' IR data.

Table 2. FT-IR data for (L΄) and It΄s complexes.

NO	Compounds	υ (N-H)	υ (C=O) Amide	υ (C=S)	υ (M–O)	υ (M–S)	υ (M–Cl)
1	$C_{12}H_9N_5O_3S$	3329	1662	1168	----	----	----
2	$[Mn(L')_2Cl_2]$	3330	1678	1199	466	416	243
3	$[Co(L')_2Cl_2]$	3325	1676	1184	474	421	258
4	$[Zn(L')_2Cl_2]$	3330	1675	1211	465	425	246
5	$[Hg(L')_2Cl_2]$	3323	1681	1213	462	430	251

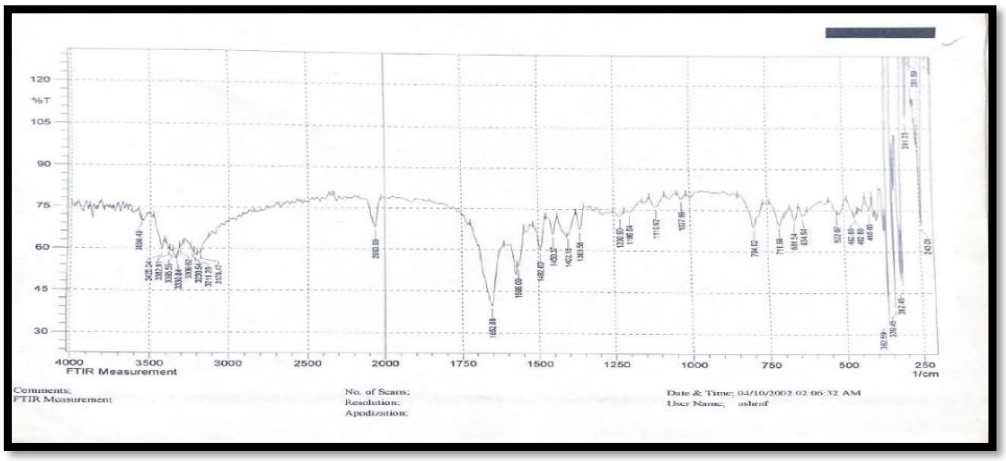


Fig. 5. Infrared spectrum of complex $[Mn(L')_2Cl_2]$.

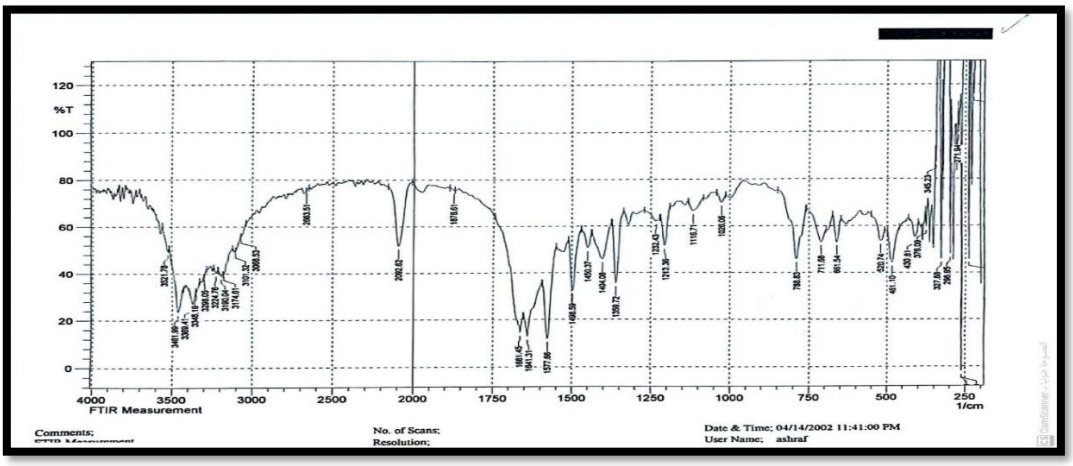


Fig. 6. Infrared spectrum of complex $[Hg(L')_2Cl_2]$

Electronic spectra

The UV-Vis transition spectra for all complexes revealed the octahedral geometric shape with the metal ion [35]. Figure (7) displays the UV-Vis spectrum of $[Mn(L')_2Cl_2]$ .Table 3 presents the electronic spectral data for complexes. Since the Zn and Hg complexes have entire d subshells, they do not exhibit any electronic transfer of the type (d - d) [28,29].

Table 3. UV data for [L'] and It's complexes.

Compounds	λ (nm)	ύ (cm^{-1})	ϵ_{max} Molar-$^{1}.cm^{-1}$	Transition Assignment	Suggested Structure	LmS.cm^2 .$mole^{-1}$	μeff (B.M)
[L']	299	33444	2372	$\pi \rightarrow \pi *$	_____	_____	_____
$[Mn(L')_2Cl_2]$	290	34482	1595	Intra-ligand			
	454	22026	963	Intra-ligand			
	716	13966	543	$^6A_1g \rightarrow {}^4T_2g(G)$	Octahedral	12.51	5.83
	865	11560	8	$^6A_1g \rightarrow {}^4T_1g(G)$			
$[Co(L')_2Cl_2]$	293	34129	1983	Intra-ligand			
	552	33557	24	^4T_1g (f) → ^4T_1g(p)	Octahedral	9.41	4.75
	888	11261	16	^4T_1g (F) → ^4A_2g(F)			
$[Zn(L')_2Cl_2]$	298	33557	2259	Intra – ligand			_____
	321	33670	1561	Intra – ligand	Octahedral	14.91	

$[Hg(L')_2Cl_2]$	301	33222	1537	Intra - ligand			____
	325	30769	1192	Intra - ligand	Octahedral	16.12	

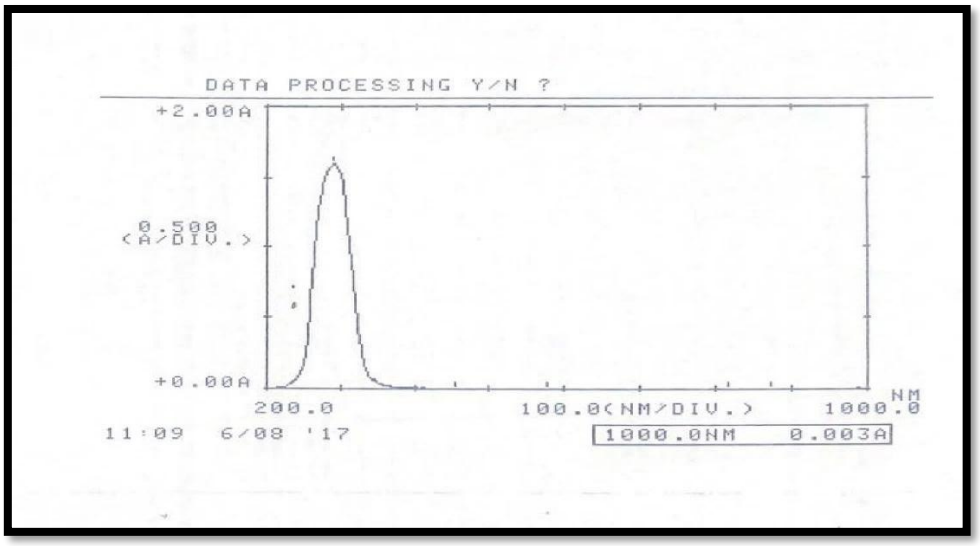


Fig. 7: UV.Vis spectrum of complex $[Mn(L')_2Cl_2]$

Magnetic moment & Molar conductivity

All molar conductivity values are tabulated in Table (3) , from the obtained values it is clear that the non-electrolyte complexes [30,31].The magnetic susceptibility values for Co(II), and Mn(II) complexes have been listed in Table 3 , Mn(II), and Co(II) complexes exhibit μeff (5.92, 4.79) B.M respectively, which may be normal values for the octahedral complexes with a high spin, Which is due to the presence of 5 lone electrons in Mn(II) ion and the presence of 3 lone electrons in Co(II) ion [32].

Antibacterial activity of the ligand and its complexes

The evaluation ligand and its metal complexes against two bacterial strains (Escherichia coli (G-) and Staphylococcus aureus (G+)) was performed using agar-well diffusion. In this method, the wells were dug in the media with the help of a sterile metallic borer with at least 6 mm centers. The recommended concentration (100 μL) of the test sample 1 mg/mL in DMSO was introduced in the respective wells. The plates were incubated immediately at 37 ºC for 24h. Activity was evaluated by measuring the diameter of inhibition zones (mm) [33,34]. The ligand [L'] and its complexes demonstrated good activity in inhibiting the growth of the bacteria under study. shown in Table 4, Figure (8).

Table (4). The prepared compounds' biological activities.

compound	*Gramm Positive*	*Gram negative*
	S. aureus	**E. coli**
[L']	7	5
[Mn(L')$_2$Cl$_2$]	10	12
[Co(L')$_2$Cl$_2$]	3	5
[Zn(L')$_2$Cl$_2$]	4	3
[Hg(L')$_2$Cl$_2$]	5	3
DMSO	0	0

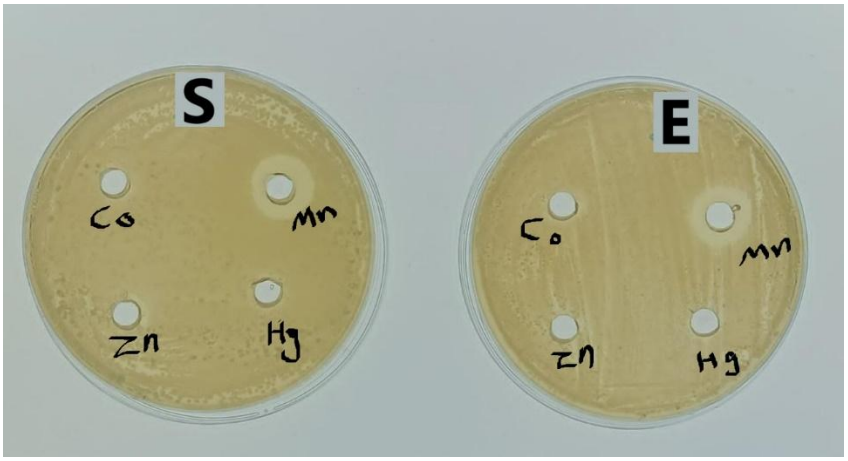


Fig. 8: Biological activity (S. aureus, E. coli,) for the complexes.

Conclusions

The suggested structural for the all complexes are depended on a molar conductivity, magnetic moment, spectroscopic research (atomic absorption, UV-Vis , FTIR) while ^{1}H – ^{13}CNMR gest for ligand [L']. It has been suggested that the behavior of the ligand [L'] is similar to bidentate in the coordination with metal ions of Mn, Co, Zn and Hg, as well as through an oxygen atom of (C=O amide) and a sulfur atom of (C=S). According to the data, the complexes have a chemical formula [M(L)$_2$Cl$_2$] (M (II) = Mn, Co, Zn, and Hg), and the octahedral geometrical structure was proposed for each complex. The effectiveness was estimated against two types of bacteria, namely (Escherichia coli, and Staphylococcus aureus) and it was found that the

complexes have more antibacterial activity than ligand so that they can be used in the future in the manufacture of pharmaceutical drugs.

Conflict of Interest

The authors declare no competing financial interests.

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تخليق ودراسة البنية والنشاط المضاد للبكتيريا لمعقدات Mn(II) و Co(II) و Zn(II) و Hg(II) مع الليكاند الجديد 4-nitro-N-(pyrimidin-2-ylcarbamothioyl) benzamide

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معلومات المؤلف
الايميل:

الخلاصة:
باستخدام 2-أمينو بيريميدين و4- نيترو بنزويل إيزوثيوسيانات، تم إنشاء ليكاند جديد 4-nitro-N - (pyrimidin-2-ylcarbamothioyl) benzamide (L'). حيث تم تحضير أربعة معقدات من الفلزات الانتقالية وهي Mn(II),Co(II),Zn(II),Hg(II) . تم تشخيص الليكاند المحضر ومعقداتها باستخدام أطياف الأشعة فوق البنفسجية والمرئية، وFT-IR و C.H.N.S و $^{1}H,^{13}CNMR$ لليكاند بينما الحساسية المغناطيسية، وقياسات التوصيلية، والامتصاص الذري فقد كانت للمعقدات. وفقا للبيانات والنتائج التي تم الحصول عليها تبين ان الليكاند يسلك كليكاند ثنائي السن من خلال ذرات S وO، وكانت الصيغة الكيميائية لكل المعقدات التي تم تحضيرها $[M(L)_2Cl_2]$ M (II) = (Mn ، Co، Zn و Hg) تم اقتراح الشكل الهندسي ثماني السطوح للمعقدات المحضرة. و أظهرت المعقدات الفلزية و الليكاند نشاطًا مضادًا للبكتيريا جيدًا ضد الإشريكية القولونية والمكورات العنقودية الذهبية.