

Synthesis , characterize and biological studies of (Cr(III) ,Mn(II)
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Abstrac

The new multidentate Schiff-base ligand (2,6-bis-[(3-phenyl thiourea-imino)-methyl]-phenol) HL and its monomeric metal complexes with Cr(III) ,Mn(II) ,Zn(II)and Cd(II) are reported. The ligand HL was derived from the condensation reaction of (2,6-diformyl-4-methyl-phenol) with (4-phenylthiosemicarbazide) in mole ratios of 1:2 . The complexes were prepared from the reaction of the corresponding metal chloride with the ligand .The ligand and its metal complexes were characterised by spectroscopic methods (FTIR, UV-Vis,¹H NMR, ¹³C. A.A), chloride content, conductance and melting point measurements. These studies revealed octahedral geometries for Cr(III), Mn(II), and Cd(II) complexes of general formulae [CrIII(L)Cl]Cl, [MnII(L)Cl] and [CdII (L)Cl] and five co-ordinate Zn(II) complex of general formula [ZnII(L)Cl]Cl .Biological activity of the ligand and its metal complexes against gram positive bacterial strain *Bacillus* (G+) and gram negative bacteria *Pseudomonase* (G-) revealed that the metal complexes become less resistive to the microbial activities as compared to the free ligand.

Keywords: Schiff-baseligand (2,6-bis-[(3-phenyl thiourea-imino)-methyl]-phenol);transition metals; structural and biological studies.

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Introduction

In principle thiosemicarbazide reacts with wide variety of aldehydes and ketones to form thiosemicarbazones, which may be used as a ligand. Compounds formed in this way are structurally interesting since both sulfur and nitrogen atoms may be involved in coordination, and many transition metal complexes of thiosemicarbazones have been reported [1]. Thiosemicarbazone moiety without substitute attached to the thion sulfur coordinates as either neutral or anionic (N,S) bidentate ligand depending on the method of complex preparation [2]. When an additional coordinating functionality is presented in the proximity of the donating centers, the ligands bind in a tridentate manner NNS, ONS. This occurs with either the neutral molecule or the mono basic anion upon loss of a hydrogen from N(2) [3]. Schiff-base metal complexes also have applications in biomedical [4, 5], biomimetic and catalytic systems [6, 7] and in supporting liquid crystalline phases [8]. Furthermore, metal complexes of chromium, manganese, nickel, copper, zinc and ruthenium with a wide variety of Schiff-bases have been used as catalysts for carbonylation, hydrogenation, hydroformylation and epoxidation reactions [9,10]. Recently ,we reported the Metal-assisted assembly of dinuclear metal (II) dithiocarbamate Schiff-base macrocyclic complexes: Synthesis and biological studies potassium 2,2`-(1*E*,1`*E*)-(1,2-diphenylethane-1,2-diylidene)bis(azan-1-yl-1-ylidene) bis (2,1phenylene)dicarbamodithioate [11]. In this paper, the synthesis and spectral charecterisation of some transition metal complexes with (2,6-bis-[(3-phenyl thiourea-imino)-methyl]-phenol) HL are reported .

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Experimental

Materials: All reagents were commercially available and used without further purification. Solvents were distilled from appropriate drying agents immediately prior to use.

Physical measurements: Melting points were obtained on a Buchi SMP-20 capillary melting point apparatus and are uncorrected. IR spectra were recorded as (KBr) disc using a Shimadzu 8400 FTIR spectrophotometer in the range 4000-400 cm^{-1} . Electronic spectra of the prepared compounds were measured in the region 250-1100 nm for 10^{-3}M solutions in DMSO at 25°C using a Shimadzu 160 spectrophotometer with $1.000\pm 0.001 \text{ cm}^{-1}$ matched quartz cell. ^1H , ^{13}C NMR, spectrum was acquired in DMSO- d_6 solution using a Bruker AMX300 MHz spectrometer with tetramethylsilane (TMS) as an internal standard for ^1H , ^{13}C NMR at AL-al-Bayt University, Jordan. Metals were determined using a Shimadzu (A.A) 680 G atomic absorption spectrophotometer. Chloride was determined using potentiometer titration method on a (686-Titro processor- 665Dosimat-Metrohm Swiss). Conductivity measurements were made with DMSO solutions using a PW 9526 digital conductivity meter.

Synthesis

Preparation of the precursor (2,6-diformyl-4-methyl-phenol): To a solution of p-cresol (10.8 g, 10 mmol) in (50 ml) glacial acetic acid, hexamethylenetetramine (28.2g,20mmol)and (30g,100mmol) of paraformaldehyde were added. The mixture was allowed to stirred continuously until the deep orange viscous solution was obtained then heated to (70-90°C).for two hrs. The solution was cooled to room temperature and concentration H_2SO_4 (10 ml) carefully added. The resulting solution was refluxed for half-hr, and then on treatment with distilled water (400 ml) a light yellow precipitate was formed, which was stored over night at (4°C).The yellow product was isolated by filtration and washed in small amount of cold methanol. More pure product was obtained by means of

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Riyadh M. Ahmad ,Hasan A. Hasan , Enaam I. Yousif and Dhuha F. Hussien

recrystallisation from toluene, Yield (%35),(5.6 g), m.p (134-132⁰C). IR data (cm⁻¹): 3349(O-H), 2970(C-H)arom, 2924 and 2791(C-H) aldehydic, 1665 (C=O), 1240 (C-O).

Preparation of the HL : Preparation of the (2,6-bis-[(3-phenyl thiourea-imino)-methyl]-phenol) .A solution of 2,6-diformyl-4-methyl-phenol (0.5g, 3.0488 mmole) in methanol(5ml) was added to 4-phenylthiosemicarbazide (1.01g, 6.0976 mmole) dissolving in methanol (5ml),and then(2-4) drops of glacial acetic acid was added slowly to the reaction mixture. The mixture was refluxed for 4 hrs, and allowed to dry at room temperature for (24) hrs. pale yellow solid metal was obtained. Yield (1.11 g , %78), m.p =87-89⁰C .IR data (cm⁻¹): 3342(O-H), 3062 (C-H)arom, 2868(C-H) iminic, 1606 (C=N). The ¹H NMR spectrum of the ligand in DMSO-d₆ showed peaks at; δH(300 MHz, DMSO-d₆): 7.34-7.56 (Ar-H); 2.78, 2.87 (N-H-2) and (N-H-4), 4.85(O-H).

General synthesis of the complexes: A methanolic solution (10 mL) of the metal salt (metal (II) salts are hydrated chloride; MCl₂.XH₂O; where: M= Cr^{III} : X=3 , Mn^{II} : X= 4 and Cd X= 2 respectively. Zinc chloride was no hydrated) was stirred into methanolic solution of the Schiff-base ligand (1 mmol) in methanol (15 mL) with (2 mmol) in methanol (15 mL) KOH. The reaction mixture was then refluxed for 2 h on a water bath until a coloured precipitate formed which was collected by filtration, washed with cold ethanol (5 mL) and dried at room temperature. Elemental analysis data, colours, and yields for the complexes are given in(Table 1).

Determination of Bacteriological Activity :Bioactivities were investigated using agar-well diffusion method [12]. The wells were dug in the media with the help of a sterile metallic borer with centers at least 24 mm. Recommended concentration (100 μL) of the test sample 1 mg/mL in DMSO was introduced in the respective wells. The plates were incubated

Synthesis , characterize and biological studies of (Cr(III) ,Mn(II)
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Riyadh M. Ahmad ,Hasan A. Hasan , Enaam I. Yousif and Dhuha F. Hussien

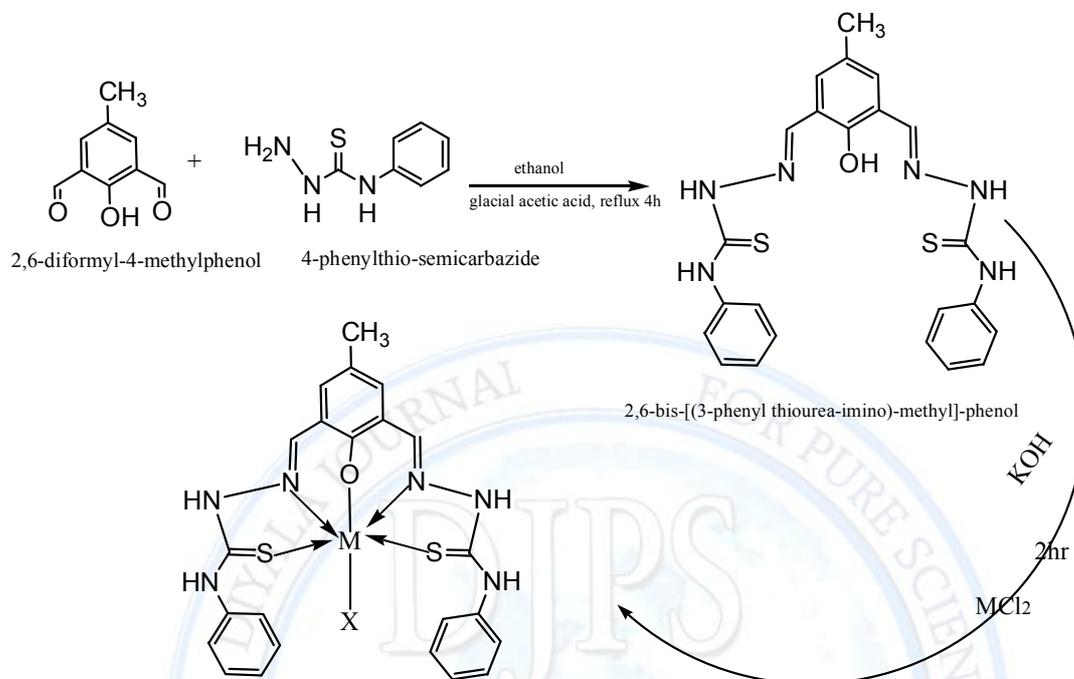
immediately at 37°C for 20 hours. Activity was determined by measuring the diameter of zones showing complete inhibition (mm). In order to clarify the role of DMSO in the biological screening, separate studies were carried out with the solutions alone of DMSO and they showed no activity against any bacterial strains. Ligand found to be potentially active against these bacterial strains compared with its complexes.

Results & Discussion

Chemistry: The condensation reaction of 2,6 diformyl-4-methyl phenol with 4-phenylthiosemicarbazide in mole ratios of 1:2 gave the Ligand (2,6-bis-[(3-phenyl thiourea-imino)-methyl]-phenol) HL in good yield. The Schiff-base ligand was characterised by elemental analysis (Table 1), IR (Table 2), UV-Vis (Table 3) spectroscopy and ¹H NMR spectrum. The di-electrolyte metal-complexes were synthesised by mixing at reflux 1 mmole of the Schiff-base ligand with 1 mmole of the appropriate metal chloride. monomeric complexes of the general formulae [M (L)Cl]Cl, [M (L) Cl] and [M (L)] Cl , (where M =Cr^{III}, Mn^{II}, Zn^{II} and Cd^{II}) was obtained (Scheme 1). The complexes are air-stable solids, soluble in EtOH, DMSO and DMF. The complexes are sparingly soluble in MeOH and not soluble in other common organic solvents. The coordination geometries of the complexes were deduced from their spectra. The analytical data (Table 1) agree well with the suggested formulae. Conductivity measurements of the complexes lie in the (23.03-6.56) cm²Ω⁻¹mol⁻¹ range, indicating their 1:1 electrolytic and non-electrolytes behavior (Table 1) [13].

Synthesis , characterize and biological studies of (Cr(III) ,Mn(II)
 ,Zn(II)and Cd(II)) complexes with Schiff-base ligand.

Riyadh M. Ahmad ,Hasan A. Hasan , Enaam I. Yousif and Dhuha F. Hussien



Scheme (1): Synthesis diagram of the Schiff-base Ligand HL and it's complexes.

IR and NMR Spectra: The important infrared bands for the ligand and its metal complexes together with their assignments are listed in Table 2. The IR spectra of the ligand shows characteristic bands at 3342, 3219 and 1614 cm^{-1} due to the $\nu(\text{O-H})$, $\nu(\text{N-H})$ and $\nu(\text{C=N})$ imine functional groups, respectively [14,15]. The IR spectra of the complexes exhibited ligand bands with the appropriate shifts due to complex formation (Table 2). The $\nu(\text{C=N})$ imine at 1614 cm^{-1} in the free Schiff-base ligand is shifted to lower frequency and observed in the range 1591-1600 cm^{-1} for the complexes. The bands are assigned to a $\nu(\text{C=N})$ stretch of reduced bond order. This can be attributed to delocalisation of metal electron density (t_{2g}) to the π -system of the ligand [16, 17], indicating coordination of nitrogen of the C=N moieties to the metal atoms [18]. Figure (1) represents the IR of the ligand and it's Zn-complex. Upon complex formation, The $\nu(\text{N-H})$ band at 3342, 3219 cm^{-1} in the free ligand is shifted and appeared at the range 3396-3225 cm^{-1} for the complexes [18]. At lower

Synthesis , characterize and biological studies of (Cr(III) ,Mn(II)
,Zn(II)and Cd(II)) complexes with Schiff-base ligand.

Riyadh M. Ahmad ,Hasan A. Hasan , Enaam I. Yousif and Dhuha F. Hussien

frequency the complexes exhibited bands around 565–651, 499–574 and 445–468 cm^{-1} which could be assigned to $\nu(\text{M-O})$ $\nu(\text{M-N})$ and $\nu(\text{M-S})$ vibration mode [16]. These bands indicated that the imine nitrogens and the thione sulphur of the ligand is involved in coordination with metal ion. The main peaks of ^1H NMR of HL are collected in the experimental section “preparation of HL and presented in Figure (3). The resonance peaks associated with the aromatic groups are observed in the range 7.34–7.56 ppm.. A resonance around 4.85 ppm equivalent to one proton assigned to the hydroxyl group. While the chemical shift at 2.78, 2.87 ppm may assigned to the secondary amine groups (*N-H-4*) and (*N-H-2*), the shift to high field may be caused by intra molecular hydrogen bonding (*N-H...N*), (*N-H...S*). The ^{13}C NMR spectrum of the ligand HL is shown in Figure (4). The spectrum reveals two sharp resonance at 180 and 155 ppm refer to the thioamide and imino carbons respectively. The sharp resonance at 22 ppm is corresponding to the methyl carbon. The shifts at the range 132.4–121.3 ppm refer to the aromatic carbon atoms. The two shifts centered at 139.4, 134.7 ppm refers to the C-N carbon atoms, finally the resonance at 117.2 ppm assigned to the C-O carbon atom.

Electronic spectra :The electronic spectra data of the ligand and its complexes are summarised in (Table 3). The UV-Vis spectrum of L exhibits a high intense absorption peaks at 282 and 302 nm, assigned to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transition respectively. The electronic spectra of the complexes Cr (III) and Mn (II) exhibit a high intensity peak around 281–295 nm related to the intra-ligand field transition. Bands exhibit around 315–338 nm assigned to the charge transfer (CT).The spectrum of the Cr (III) complex exhibited band characteristic of octahedral Cr (III) complexes [19–22]. The electronic spectrum of the Mn (II) complex shows band which can be attributed to $^6\text{A}_{1g}(\text{S}) \rightarrow ^4\text{T}_{1g}(\text{G})$ transition, corresponding to octahedral geometry [19, 23]. The spectrum of the Zn(II) and Cd(II) complexes exhibited bands assigned to ligand $\pi \rightarrow \pi^*$ and L \rightarrow M charge transfer [19, 25].

Synthesis , characterize and biological studies of (Cr(III) ,Mn(II)
,Zn(II)and Cd(II)) complexes with Schiff-base ligand.

Riyadh M. Ahmad ,Hasan A. Hasan , Enaam I. Yousif and Dhuha F. Hussien

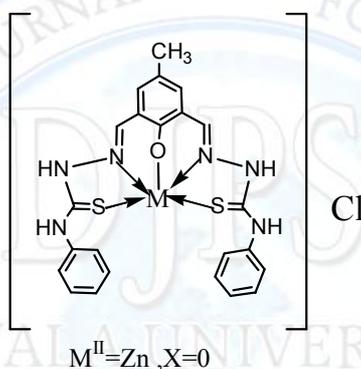
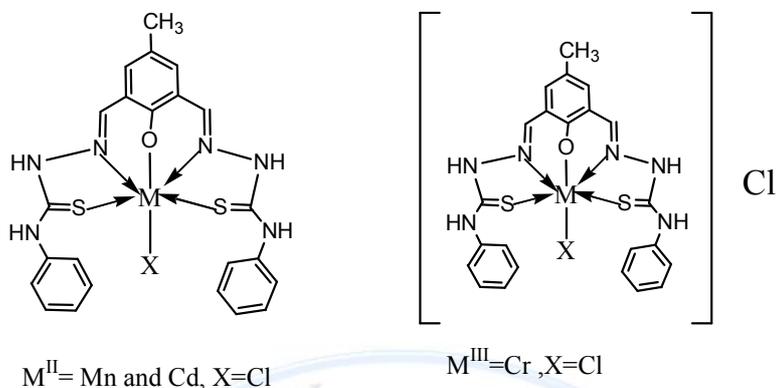
The Cd(II) complexe adopt octahedral geometries [26], while a five-coordinate structure is suggested for the Zn(II) centre [27, 28].

Antimicrobial activity: The free Schiff-base ligand and its metal complexes were screened against *Bacillus* (G+) and *Pseudomonase* (G-) to assess their potential as an antimicrobial agent by disc diffusion method. The measured zone of inhibition against the growth of various microorganisms is listed in Table 4. It is found that the ligand has higher antimicrobial activity than its metal complexes. This can be explained as follows: The biological activity of the Schiff-base ligand is related to the imine moiety, which plays a key role in the inhibition of the tested bacteria. The lower antimicrobial activity of the metal complexes compared with that in the ligand may be due to the strong interaction between the imine moieties and the metal ions. Such interaction will reduce the activity of the imine moiety in the inhibition.

Conclusion: In this paper, the synthesis and coordination chemistry of some monomeric metal complexes derived from the Schiff base HL are investigated. The complexes were prepared by mixing at reflux 1 mmole of the Schiff-base ligand with 1 mmole of the appropriate metal chloride. Complexes of the general formulae $[M(L)Cl]Cl$, $[M(L)Cl]$ and $[M(L)]Cl$ (where M = Cr(III), Mn(II), Zn(II) and Cd(II)) was obtained. Physico-chemical analysis indicated the formation of four coordinate dicationic metal complexes. Biological activities revealed that the ligand has higher antimicrobial activity than its metal complexes.

Synthesis , characterize and biological studies of (Cr(III) ,Mn(II) ,Zn(II)and Cd(II)) complexes with Schiff-base ligand.

Riyadh M. Ahmad ,Hasan A. Hasan , Enaam I. Yousif and Dhuha F. Hussien



Scheme(2): Proposed structures of complexes

Synthesis , characterize and biological studies of (Cr(III) ,Mn(II)
,Zn(II)and Cd(II)) complexes with Schiff-base ligand.

Riyadh M. Ahmad ,Hasan A. Hasan , Enaam I. Yousif and Dhuha F. Hussien

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Synthesis , characterize and biological studies of (Cr(III) ,Mn(II)
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Riyadh M. Ahmad ,Hasan A. Hasan , Enaam I. Yousif and Dhuha F. Hussien

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Synthesis , characterize and biological studies of (Cr(III) ,Mn(II)
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Riyadh M. Ahmad ,Hasan A. Hasan , Enaam I. Yousif and Dhuha F. Hussien

Table1 Colours, yields, elemental analyses, and molar conductance values.

Compound $^1\text{mol}^{-1}$	Colour	Yield (%)	m.p	Found (Calcd.) (%)		$\Lambda_M(\text{cm}^2\Omega^{-1})$
				M	Cl	
HL	yellow	78	87-89	-	-	-
[Cr ^{III} (L) Cl] K	green	75	215	8.11 (8.90)	11.29 (12.13)	38.03
[Mn ^{II} (L) Cl]	brown	73	320	8.84 (9.95)	5.20 (6.42)	15.66
[Zn ^{II} (L) Cl]	yellow	75	205	10.78 (11.63)	6.07 (6.30)	36.56
[Cd ^{II} (L) Cl]	yellow	54	265	17.73 (18.44)	5.43 (5.82)	8.12

**Synthesis , characterize and biological studies of (Cr(III) ,Mn(II)
,Zn(II)and Cd(II)) complexes with Schiff-base ligand.**

Riyadh M. Ahmad ,Hasan A. Hasan , Enaam I. Yousif and Dhuha F. Hussien

Table 2. IR frequencies (cm^{-1}) of the compounds.

Compound	$\nu(\text{N-H})$	$\nu(\text{C=N})_{\text{imin}}$	$\nu(-\text{N-C-S})$	$\nu(\text{C=S})$	$\nu(\text{N-N})$	$\nu(\text{M-O})$	$\nu(\text{M-N})$	$\nu(\text{M-S})$
HL	3342,3219	1614	1446	867,1342 (w)	1029	-	-	-
[Cr ^{III} (L) Cl] Cl 468	3396,3366	1600		1475	848,1398 (w)	1035	651	574
[Mn ^{II} (L) Cl] 445	3300,3224	1597		1496	827,1355 (w)	1078	565	499
[Zn ^{II} (L) Cl] 464	3295,3138	1598		1490	831,1357 (w)	1080	617	569
[Cd ^{II} (L) Cl] 456	3225 ,3188	1591		1520	822,1375 (w)	1079	626	552

**Synthesis , characterize and biological studies of (Cr(III) ,Mn(II)
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Riyadh M. Ahmad ,Hasan A. Hasan , Enaam I. Yousif and Dhuha F. Hussien

Table 3: U.V-Vis spectral data in DMSO solutions.

Compound	Band position (λ nm)	Extinction coefficient $\epsilon_{max}(\text{dm}^3 \text{mol}^{-1}\text{cm}^{-1})$	Assignments
HL	282	3915	$\pi \rightarrow \pi^*$
	302	3804	$n \rightarrow \pi^*$
[Cr ^{III} (L) Cl] Cl	295	870	$\pi \rightarrow \pi^*$
	315	722	CT
[Mn ^{II} (L) Cl]	670	577	${}^4A_{2g} \rightarrow {}^4T_{2g}^{(F)} (m1) (10 Dq)$
	281	690	$\pi \rightarrow \pi^*$
[Zn ^{II} (L) Cl]	338	448	CT
	608	122	${}^6A_{1g}^{(S)} \rightarrow {}^4T_{1g}^{(G)}$
[Cd ^{II} (L) Cl]	277	614	$\pi \rightarrow \pi^*$
	310	523	CT
[Cd ^{II} (L) Cl]	265	930	$\pi \rightarrow \pi^*$
	333	435	CT

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 ,Zn(II)and Cd(II)) complexes with Schiff-base ligand.

Riyadh M. Ahmad ,Hasan A. Hasan , Enaam I. Yousif and Dhuha F. Hussien

Table 4: Antibacterial activities of the synthesised Schiff-base and metal complexes.

Compounds	<i>Bacillus</i> (G+)	<i>Pseudomonase</i> (G-)
HL	+++++	++++
[Cr ^{III} (L)Cl] Cl	+++	++
[Mn ^{II} (L) Cl]	+	+++
[Zn ^{II} (L)]Cl	+	++
[Cd ^{II} (L) Cl]	++	++

Synthesis , characterize and biological studies of (Cr(III) ,Mn(II) ,Zn(II)and Cd(II)) complexes with Schiff-base ligand.

Riyadh M. Ahmad ,Hasan A. Hasan , Enaam I. Yousif and Dhuha F. Hussien

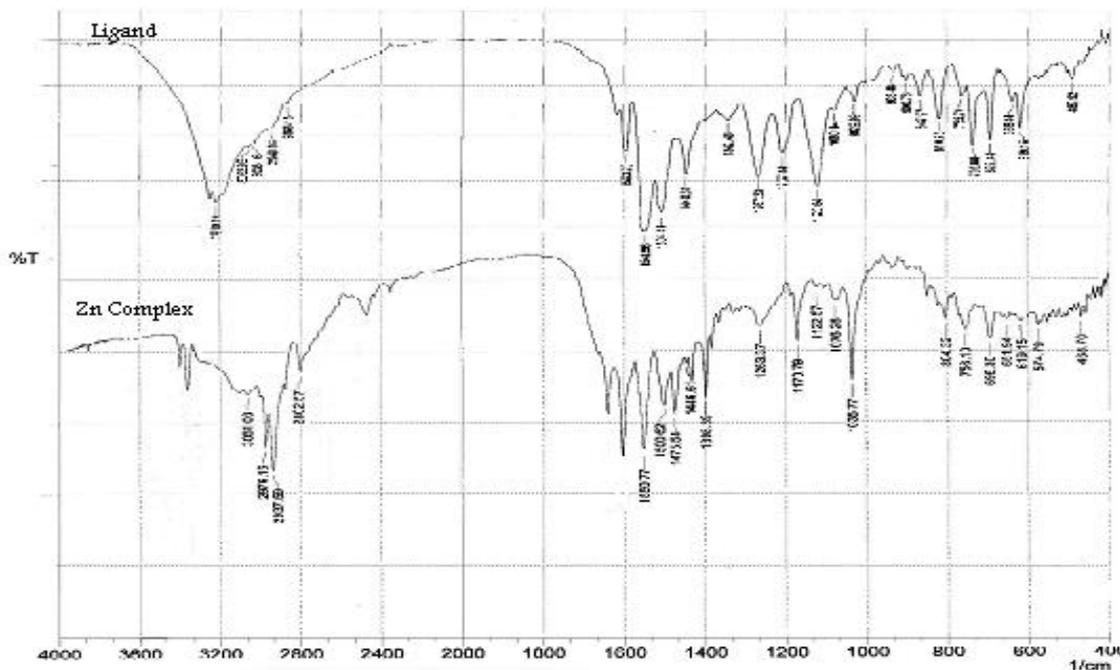


Figure 1: IR spectra for the ligand and Zn-complex

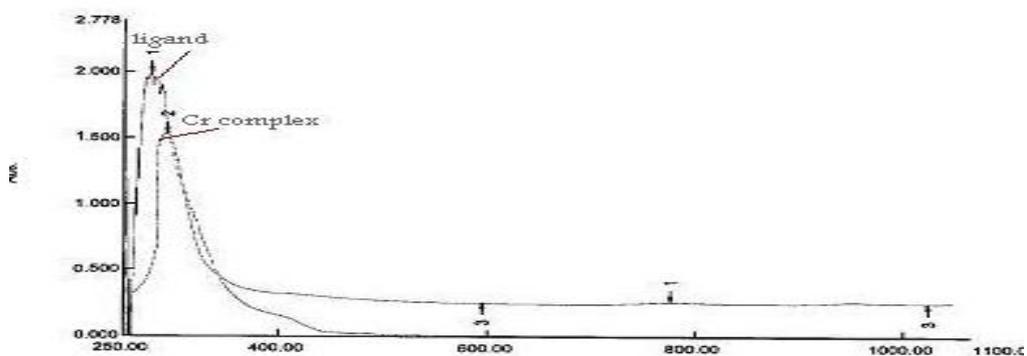


Figure 2: Electronic spectrum of the ligand and Cr - complexes.

Synthesis , characterize and biological studies of (Cr(III) ,Mn(II)
,Zn(II)and Cd(II)) complexes with Schiff-base ligand.

Riyadh M. Ahmad ,Hasan A. Hasan , Enaam I. Yousif and Dhuha F. Hussien

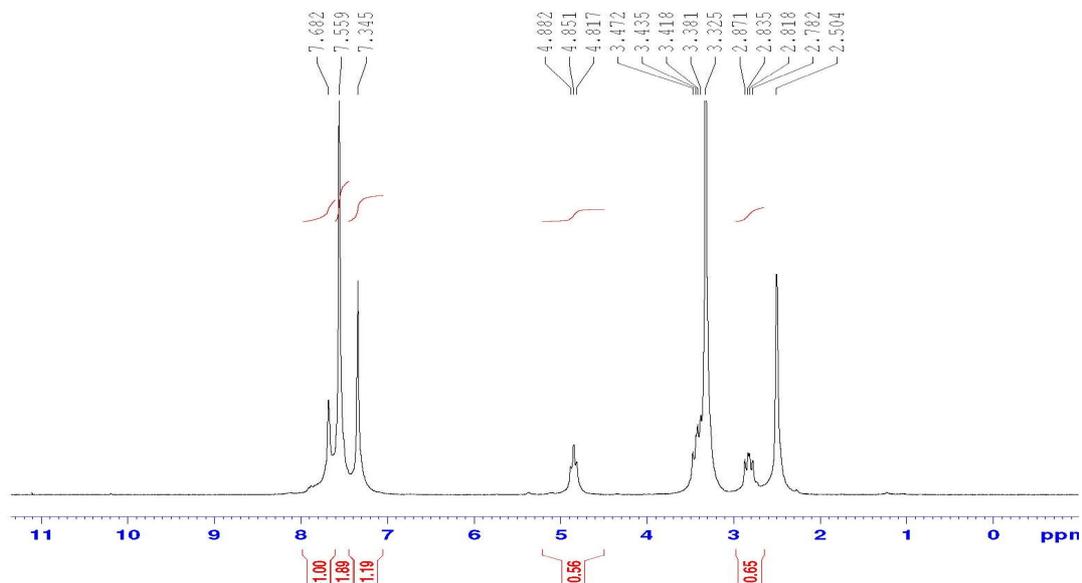


Figure 3: ¹H-NMR spectrum of the ligand HL in DMSO-d₆ solution.

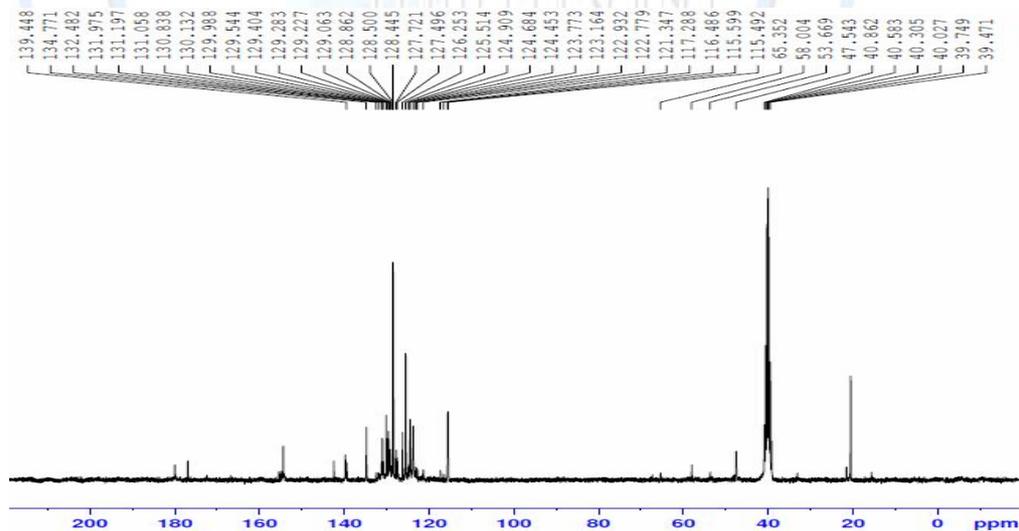


Figure 4: ¹³C spectrum of the ligand HL in DMSO-d₆ solution

Synthesis , characterize and biological studies of (Cr(III) ,Mn(II)

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Riyadh M. Ahmad ,Hasan A. Hasan , Enaam I. Yousif and Dhuha F. Hussien

معقدات احادية النواة الجديدة

مع قاعدة شف تحضير ، تشخيص و دراسة الفعالية البايولوجية.

رياض محمود احمد ،حسن احمد حسن، انعام اسماعيل يوسف و ضحى فاروق حسين

جامعة بغداد

كلية التربية- ابن الهيثم- قسم الكيمياء

P.O.4150 العراق - بغداد - الاعظمية -

الخلاصة

تضمن البحث تحضير الليكاند الجديد قاعدة شف

[2,6-bis-[(3-phenyl thiourea-imino)-methyl]-phenol] (HL)

المشتق من

2,6-diformyl-4-methyl-phenol و 4-phenylthiosemicarbazide

ثم مفاعلة مع بعض العناصر الفلزية باستخدام الميثانول وسطا للتفاعل وبنسبة (1:1) حيث تكونت معقدات جديدة

ذات الصيغ العامة:

[M (L)Cl] Cl

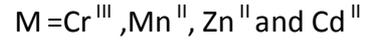
[M (L)Cl]

[M (L)Cl]

Synthesis , characterize and biological studies of (Cr(III) ,Mn(II)
,Zn(II)and Cd(II)) complexes with Schiff-base ligand.

Riyadh M. Ahmad ,Hasan A. Hasan , Enaam I. Yousif and Dhuha F. Hussien

حيث:



شخصت جميع المركبات بالطرق الطيفية التالية (الأشعة تحت الحمراء والأشعة فوق البنفسجية – المرئية ومطيافية الامتصاص الذري للعناصر ^{13}C ، ^1H NMR، ومحتوى الكلور ودرجات الانصهار) ، مع قياس التوصيلية المولارية الكهربائية والفعالية البيولوجية.، من النتائج أعلاه كان الشكل الفراغي المقترح لمعقدات الكروم ، المنغيز والكاديوم ثماني السطوح بينما الزنك يتخذ شكل خماسي التناسق.

