Article

Preparation and Characterization of Schiff Base Ligand Complexes Derived from Dimedone with Urea

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Abstract

Eight complexes of this group were created in this work by reacting the novel ligand that was produced from the reaction of dimedon with urea with the chlorides of trivalent elements (Iron and Chromium) and divalent transition elements (Manganese, Cobalt, Nickel, Copper, Platinum, and Cadmium). These complexes had unique hues, melting temperatures, and solubility's based on the diagnostic data, it was discovered that the ligand**1,5,5-carbamoylimino-3,3-dimethylcyclohexylidene urea (L1)** It attaches to the metal ion by two amine groups and functions as a Bi-dentate ligand . IR, UV-visible, Molar electrical conductivity, and Magnetic susceptibility Spectra all suggested Paramagnetic characteristics for the complexes, with the exception of Zinc and Cadmium are Dimagnetic, and Molar conductivity revealed that all the complexes are non-Electrolytic, with the exception of Iron and Chromium. These data suggested that the complexes were Octahedral in shape. Elemental analysis was used to identify the molecular formula of the complexes that made up the group, and the elemental percentages agreed with the suggested molecular formula. The outcomes demonstrated that the complexes' biological activity against pathogenic bacteria exhibited a range of efficacy. When ethanol was used as a test substance for antioxidant efficacy, the results varied in terms of efficacy and were compared to gallic acid using DPPH as a free radical, yielding differing IC_{50} values.

Keywords: Dimedone, Urea, Schiff Base Complex, IC_{50}, Octahedral

الخلاصة

لقد تم في هذه الدراسة تحضير ثمان معقدات هذه المجموعة تم تحضيرها من خلال مفاعلة الليكاند الجديد المحضر من تفاعل (الدايميدون مع اليوريا) مع كلوريدات العناصر الانتقالية ثنائية التكافؤ (المنغنيز و الكوبلت و النيكل و النحاس والخارصين و الكادميوم) وثلاثية التكافؤ (الحديد والكروم). وقد تميزت هذه المعقدات بألوان مميزة ودرجات انصهار وقابلية ذوبان مختلفة، ومن النتائج التشخيص التي تم الحصول عليها وجد ان الليكند 1,5-5-carbamoylimino-3,3-dimethylcyclohexylidene urea (L1).

التي تتصرف كليكندات ثنائية السن مع الايون الفلزي حيث ترتبط به من خلال مجموعتي امين. وتم أقتراح الشكل ثماني السطوح لمعقدات وذلك من خلال أطياف الاشعة تحت الحمراء و الاشعة فوق البنفسجية – المرئية والتوصيلية الكهربائية المولارية والحساسية المغناطيسية وجميعها أشارت ذات صفات بارا مغناطيسية ما عدا

الخارصين والكادميوم ذات صفات دايا مغناطيسية والتوصيلية المولارية قد بينت أن جميع المعقدات غير ألكتروليتية ماعدا الحديد والكروم. وتم تحديد الصيغة الجزيئية للمعقدات المتكونة للمجموعة من خلال التحليل الدقيق للعناصر ،حيث تطابقت النسب المئوية للعناصر مع الصيغة الجزيئية المقترحة . وأظهرت النتائج قياس الفعالية البيولوجية ضد البكتريا المرضية للمعقدات فعالية متفاوتة .وأظهرت نتائج قياس الفعالية لمضادات الاكسدة باستخدام الايثانول فعالية متفاوتة وقورنت مع حامض جاليك باستخدام مادة DPPH كجذر حر وقد اعطت قيم (IC50) مختلفة .

1- Introduction

Schiff bases are nitrogenous organic compounds similar to Aldehydes and Ketones where the carbonyl group (C=O) has been replaced by an Azomethin group [1], also called Amines, and are named Schiff bases after the chemist Hugo Schiff who performed the reaction of an Aldehyde or Ketone with a primary amine [2] Schiff bases have received great and wide attention in terms of Chemistry, Application and biology.This is because they are intermediates for the preparation of many derivatives. Schiff bases have been prepared by the Condensation reaction of an aldehyde or ketone with a primary amine with the omission of a water molecule. These reactions are the most common in the preparation of Schiff bases. [3]The field of Coordination Chemistry has been active for more than a century now. The 19th century saw the beginning of efforts to list and Characterize what are now known as Coordination Compounds. In 1857, Wolcott Gibbs and Frederick Genth published their work on what they referred to as Ammonia and Cobalt bases, drawing attention to a class of salts. [4] In the latter half of the 20th century, Coordination Chemistry gained a broad chemical field due to its rapid development in the practical aspect of preparing Complex Coordination Compounds (Coordination Compound) and interpreting their structure, which has played a large and important role in a number of fields that have been tested and confirmed their importance at present, Including Agriculture, Industry, and Medicine[5].

Coordination complexes consist of an atom or ion that accepts a pair of electrons called an acceptor and an atom or ion that provides that pair of electrons called a donor called a ligand, which is part of a neutral molecule such as CO", H_2O and NH_3", or part of an ion such as $NH_2CH_2COO^-$ and CO_3^-. The acceptor binds to a number of donor atoms that may be identical or dissimilar and the resulting compound may be a positive ion, a negative ion or a neutral molecule. [6-8] Transition elements are not the only elements in the reaction that can form complexes; Symmetrical elements can also form complexes by the interaction of metal ions with either organic or inorganic ions or molecules known as ligands.[9] What distinguishes organic ligands is that they contain active groups whose atoms are electron donors and behave like Lewis bases, and the most famous of these active groups are (C=S, N=O, N=N, NH_2, C=N), in addition to containing active groups that also have a role in increasing the colour intensity of these ligands and their complexes. [10]. The coordination bond is formed by donating an electron pair from a Lewis base ligand to another receptor containing an empty orbital (central metal) Lewis's acid and the new bond formed is called a coordination bond. [11-12].

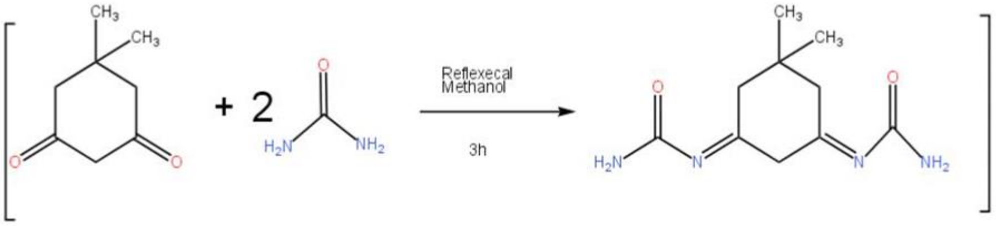
2 - Materials and Methods

1- Materials and interfaces

The materials used in this study were B.D.H Elemental Chlorides with a purity of 99%.Fluke and C.D.H. solvents had purities ranging from 98-99.9%. As for the instruments used, the FT-IR Spectrum was measured by Bruker and the Uv Spectrum was measured by Shimiad ZU Uv-Visible Spectro Photometer. And magnetic susceptibility measurement Balance Susceptibility Magnatic type device The measurement was made at the Ministry of Science and Technology - Environmental and Water Technology Research Department.

2- Preparation of ligand L1 :

(1.40gm, 1mmol) of dimedon was added to (1.20gm, 2mmol) of (Urea) with (10) drops of Glaice acetic acid as a catalyst and dissolved in 25ml of absolute ethanol of 99.9% purity and placed together in a 100ml round bottom flask with continuous stirring. It is placed on heating to a temperature of (80) degrees Celsius with the use of a stirrer to ensure the dissolution of the substances and left on heating for (3 h) using retrograde rectification to form yellow crystals. The formed crystals were filtered and recrystallized using ethanol and the melting point (156-152)℃ as in the following scheme:



Scheme 1: 1,5,5-(carbamoylimino)-3,3-dimethylcyclohexylidene)Urea

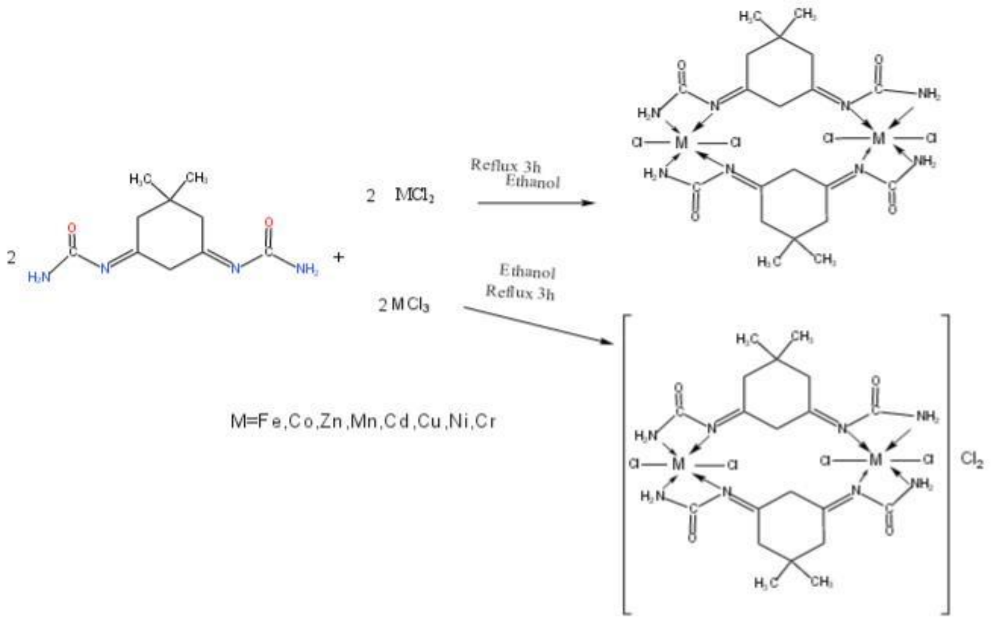
3- Preparation of ligand (L1) complexes:

Ligand complexes were prepared from the reaction of previously prepared ligand with metal salts using the following steps:

In a 100 ml round bottom flask (0.520 g, 2 mmol) of **L1** dissolved in 20 ml of absolute ethanol to dissolve the L1 was placed (0.474 g, 2 mmol) of Cobalt Chloride salt ($CoCl_2.6H_2O$) dissolved in 20 ml of absolute ethanol with constant stirring. The dissolved ligand (L1) was then mixed with the Cobalt Chloride salt. The mixture was heated using reflux heating with continuous stirring for 3h. It is heated to (70) degrees clsius. L1 was added to the salt in a ratio of (2:2) and a dark brown precipitate was obtained, which was filtered and recrystallized using ethanol and weighed and the percentage of the resulting complex was (83%), then the reaction was confirmed by thin layer chromatography (TLC). The structure of the complexes was prepared by dissolving Mn(II), Fe(III), Cr(III), Ni(II), Zn(II), and Cd(II) chlorides. The same method is shown in.

Table 1: Amount of metal salt used in complex preparation, molecular formulas of the prepared complexes, their weights, proportion and colour of the resulting complex.

NO	Metal salt	Wight(g)	Complex	Color	yield	m.p℃
1.	$CdCl_2.H_2O$	0.402	$[Cd_2L_1Cl_2]$	White	46%	206-209
2.	$CoCl_2.6H_2O$	0.474	$[Co_2L_1Cl_2]$	Dark blue	83%	200-204
3.	$CrCl_3.6H_2O$	0.532	$[Cr_2L_1Cl_4]Cl_2.6H_2O$	Green	43%	197-200
4.	$CuCl_2.2H_2O$	0.340	$[Cu_2L_1Cl_2]$	Light green	65%	197-199
5.	$FeCl_3.6H_2O$	0.324	$[Fe_2L_1Cl_4]Cl_2.6H_2O$	Dark brown	50%	210-215
6.	$MnCl_2.4H_2O$	0.394	$[Mn_2L_1Cl_2].2H_2O$	Black	52%	201-205
7.	$NiCl_2.6H_2O$	0.474	$[Ni_2L_1Cl_2]$	Dark green	45%	203-209
8.	$ZnCl_2$	0.272	$[Zn_2L_1Cl_2]$	Dark yellow	81%	202-209



Scheme 2: Preparation of metal complexes

3- Results and discussion.

3.1. Infrared Spectroscopy (IR):

The Characterization of ligand as the main preparation in the preparation of metal complexes by Infrared Spectroscopy and its comparison with the Characterization of Dimedon. It resulted in the appearance of absorption bands of the C=O group located at a frequency(1695 cm^{-1}), which indicates the presence of a carbonyl ketone group within the ring.[13,14] In the prepared ligands, the appearance of absorption bands of the C=N group was observed at frequencies (1678)cm^{-1} ligand.[15,16] As well as the appearance of vibrational absorption bands for the NH2,NH group, the frequencies appeared within the range of (3348 cm^{-1}) and (3444) cm^{-1}respectively[17]. As well as for the C=O group within the range of (1463 cm^{-1}), which indicates the presence of the amide group of the ligand [18].And measured for the rest of the prepared complexes as shown in Table (3) The following results were obtained that prove the formation of ligand and its complexes. The appearance of absorption bands of the C=N group, which showed frequencies in the range of (1620-1678) cm^{-1}. [19]. The appearance of stretching vibration absorption bands for the (NH) and (NH_2) groups, respectively, which showed frequencies in the range (3305-3479) cm^{-1} [20]. The appearance of C=O absorption bands in the range of (1384 – 1519) cm^{-1}, which indicates the presence of the amide group of ligand and complexes. [21]. Absorption bands appeared for the(M-N) group with frequencies in the range (582 - 594) cm^{-1}, which confirms the bonding of the ligand with the metal by these atoms and the occurrence of coordination. And the appearance of absorption absorption bands (M-Cl), whose frequencies are in the range (426 - 599) cm^{-1}, which indicates the coordination of ligand with the metal .[22](M-O) absorption bands with frequencies in the range of (840-777)cm^{-1} appeared for only three complexes (CrL1, CoL1, FeL1), which indicates the presence of a water group attached to ions, for complexes (Chromium, Iron and Cobalt) in the range (3415-3430 - 3377) Cm^{-1}, respectively, which is the absorption of the (O-H) atoms of a water molecule attached to ions, which is a broad intensity beam. [23]

Table 2: Shows the FT-IR absorption band values for ligand and complexes

NO	Complexes	v-NH	NH_2	C=N	C=O	H_2O	M-O	M-N	M-Cl
1.	Dimdon	-------	-----	------	1695 m-sh-	-----	-----	------	--------
2.	L1	3444 sh	3348 Sh	1678 s-b	1463 s-sh	-----	----	-----	------
3.	$C_{20}H_{36}Cr_2N_8O_4Cl_6$	3415 w	3305 w	1635 S-b	1479 s-b	3425 b	840 s	775 w	509 b
4.	$C_{20}H_{36}Mn_2N_8O_4Cl_4$	3492 s-b	3392 s-b	1550 s-b	1456 s-sh	3392 b	-----	624 m-sh	518 m-sh
5.	$C_{20}H_{36}Fe_2N_8O_4Cl_6$	3430 s-sh	3321 Sh	1633 b	1462 s-w	3910 b	827 w	688 s-b	426 m-b
6.	$C_{20}H_{36}Co_2N_8O_4Cl_4$	3377 b	3367 B	1639 B	1477 m-sh	------	777s	594 s	530 S
7.	$C_{20}H_{36}Ni_2N_8O_4Cl_4$	3447 s-sh	3437 m-b	1673 s-b	1463 s-sh	-----	-----	626 s-b	518 s-b
8.	$C_{20}H_{36}Cu_2N_8O_4Cl_4$	3456 Sh	3392 s-sh	1635 S	1475 s-sh	----	-----	582 s	528 s-b
9.	$C_{20}H_{36}Zn_2N_8O_4Cl_4$	3464 s-sh	3354 s-sh	1637 S-b	1494 s-sh	-----	-----	615 s-sh	524 s-sh
	$C_{20}H_{36}Cd_2N_8O_4Cl_4$	3479 sh	3417 sh	1579 m-sh	1469 Sh	----	------	771 s	599 S

S=Small, Sh=Sharb, W=weak , b=broad, m=middle

4-Ultra violet – visible Spectroscopy of dimedon and prepared ligand

The UV-Visible Spectrum of dimedon showed a broad absorption peak at a wavelength. A broad absorption peak at a wavelength of (271) nm, (36900) cm^{-1} is indicative of the $\pi \rightarrow \pi^*$ electronic transition. As in Figure 3-19. The UV Spectrum of the prepared ligand also showed absorption peaks at (305) nm, (32786) cm^{-1}. Which is due to the n→π* electronic transition and an absorption peak at (289) nm, (34602) cm^{-1} which is due to the π→π* electronic transition. [24-26]. The prepared complexes were also measured and gave the results shown in Table (3).

4.1Chromium complex $[Cr_2(L1)_2Cl_4]Cl_2.6H_2O$

The Electronic Spectrum of the Cr(II) complex showed absorption peaks at wavelengths of clear intensity, the first at (813)nm, (12300) cm^{-1} which is due to the d-d transition of the $^4A_2g\rightarrow {}^4T_2g$ type as well as at (745)nm, (13422) cm^{-1} for the $^4A_2g_f\rightarrow{}^4T_2g_f$, a wavelength at (542)nm, (18450) cm^{-1} for the $^4A_2gf\rightarrow{}^4T_2gp$ a wavelength at (381)nm, (26246) cm^{-1} for the M→LCT, n→π* as well as a wavelength at (261)nm, (38314) cm^{-1} that belongs to the π→π* transition. This is consistent with the Octahedral geometry. [24]

4.2Manganese complex $[Mn_2(L1)_2Cl_4].2H_2O$

The Electronic Spectrum showed absorption peaks at distinct wavelengths, the first at (915) nm, (10928) cm^{-1} belonging to the $^6A_2g\rightarrow{}^4T_2g_f$ d-d transition, the second at (542) nm, (18450) cm^{-1} belonging to the $^6A_1g\rightarrow{}^4T_1g_f$ transition, and the third at (273) nm, (36630) cm^{-1} belonging to the n→π* transition, Intra-ligand, which is consistent with the Octahedral geometry of Manganese complexes. This is because is one of the most stable oxidation states. Because the orbital ($3d^5$) is half-filled with an indivisible single state (^6A_1g), electron transitions occur from stable ^6A_1g to excited ^4G, ^4D, and ^4P[26].

4.3$[Fe_2(L1)_2Cl_4]Cl_2.6H_2O$

The UV Electronic Spectrum showed absorption peaks at wavelength (813)nm, (12300)cm^{-1} belonging to the $^3A_2g\rightarrow{}^3T_2g$ electronic transition. As well as at (745)nm, (113422) cm^{-1} which is due to the $^3A_2g\rightarrow{}^3T_1g$ electronic transition. And at (270)nm, (37037) cm^{-1} which had a long and clear peak indicating the π→π* and intra-ligand transition as well as the appearance of absorption peaks at (224)nm, (44642) cm^{-1} which is due to the n→π* electronic transition. This is consistent with the Octahedral geometry of the Iron complexes. [25]

4.4Cobalt complex $[Co_2(L1)_2Cl_4]$

Absorption peaks appeared at wavelength (890nm) (11253cm^{-1}) due to the $^4T_1g\rightarrow{}^4A_1g$ transition. As well as at (267) (37453 cm^{-1}) which is due to the π→π* transition and at (216 nm) (4296 cm^{-1}) which is due to the n→π* electronic transition. This is consistent with the Octahedral geometry of Cobalt complexes. [27].

4.5Nickel complex $[Ni_2(L1)_2Cl_4]$

Showed absorption peaks (872)nm, (11274) cm^{-1}, (772)nm, (719) nm, (14409) cm^{-1} which are due to electronic transitions are: $^3A_2g\rightarrow{}^3T_2g$, $^3A_2g_f\rightarrow{}^3T_1g_f$,

$^3A_2g_f \rightarrow {}^3T_1g_p$. Also at (542)nm, (18450) cm^{-1} wavelength, peaks at (26525) cm^{-1} (M→LCT n→π*) and absorption peaks at (283)nm, (135714) cm^{-1}belonging to the π→π* transition. This is consistent with the Octahedral geometry. [27].

4.6Copper complex [$Cu_2(L1)_2Cl_4$]

Showed absorption peaks (881)nm, (11274) cm^{-1} and (542)nm, (18450) cm^{-1} which are due to the electronic transitions $^2Eg \rightarrow {}^2T_2g$ and $^2E_2g \rightarrow {}^2T_1g$. which belongs to orbital d. Also at(291) nm ,(34364) cm^{-1} which is due to the n→π* transition. As well as peaks at (209)nm, (47846) cm^{-1} which are due to the π→π* transition. This is consistent with the deformed Octahedral geometry. [24,28]

4.7Zinc complex [$Zn_2(L1)_2Cl_4$]

Absorption peaks appeared at (364)nm, (27472) cm^{-1} due to π→π* and Intra-ligand, and at (279)nm, (35842) cm^{-1}due to n→π* and Intra- ligand and no absorption peak appeared after (364)nm. This is because $3d^{10}$ orbitals are saturated so there are no other transitions. Therefore, based on elemental analysis, magnetic susceptibility, and molar conductivity, an Octahedral morphology of Zinc complexes was proposed and the metal content and chlorine content were measured. [28].

4.8Cadmium complex [$Cd_2(L1)_2Cl_4$]

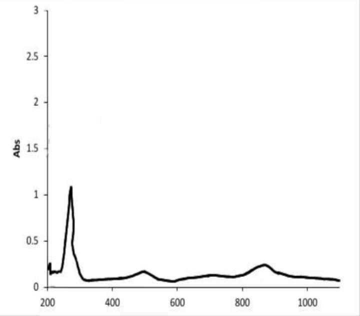
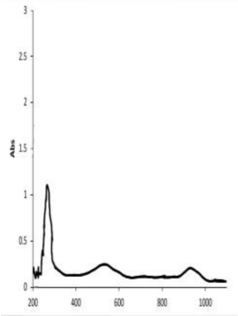
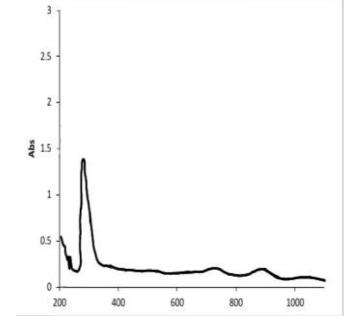
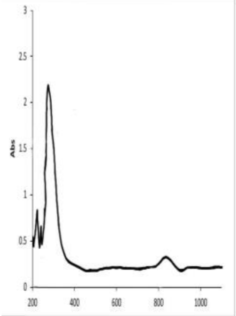
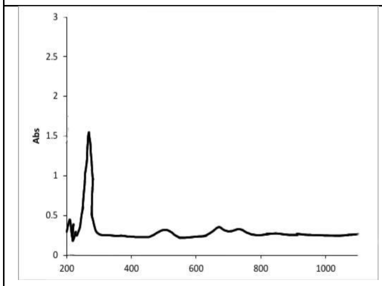
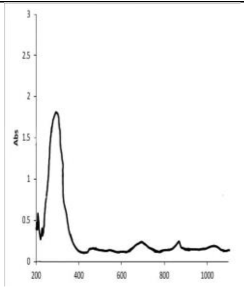
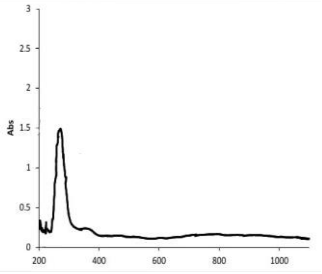
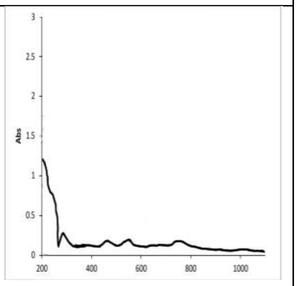
Absorption peaks appeared at wavelength (542)nm, (18450) cm^{-1}which is due to the ligand-intra electronic transition. Also at (497)nm, (20120) cm^{-1}which is also due to the Intra-ligand transition, (293)nm, (34129) cm^{-1} , which is due to the M→LCT transition, (289)nm, (34602) cm^{-1}, which is due to the n→π* transition, and (225)nm, (4444) cm^{-1} which is due to the π→π* transition. No absorption is seen above (542) nm because orbital d is filled with Cadmium ($4d^{10}$). Therefore, an Octahedral shape was proposed based on elemental microanalysis measurements, molar conductivity, metal ratio measurement, and chlorine content measurement, which was consistent with the octahedral geometry as published in the literature. [29,30]

Table (3) shows the UV Spectral data of the prepared ligand and Its complexes

NO	Compound	ʎmax	Abs	γcm^{-1}	Emax Mol^{-1}cm^{-1}	Electronic Transition type	Shape
1.	Dimedon	271	-----	36900	3311	$\pi \rightarrow \pi *$	-----
2.	L_1	305 289	-----	32786 34602	32786 34602	$n \rightarrow \pi *$ $\pi \rightarrow \pi *$	----
3.	$C_{20}H_{48}Cr_2N_8O_{10}Cl_6$	813 745 542 381	0.006 0.005 0.001 0.007	12300 13422 18450 26246	60 50 10 70	$^4A_2g \rightarrow {}^4T_2g$ $^4A_2g \rightarrow {}^4T_1g$ $^4A_2g \rightarrow {}^4T_1g_f$ M →LCT,$n \rightarrow$	Octa-headral

		261	1.255	38314	1255	$\pi *$ $\pi \rightarrow \pi *$	
4.	$C_{20}H_{40}Mn_2N_8O_6Cl_4$	915 542 273	0.010 0.008 2.582	10928 18450 36630	10 80 2582	$^6A_2g \rightarrow {}^4T_2g$ $^6A_1g \rightarrow {}^4T_1g$ Intra-Ligand , $n \rightarrow \pi *$	Octa-headral
5.	$C_{20}H_{48}Fe_2N_8O_{10}Cl_6$	813 745 270 224	0.009 0.009 0.761 0.106	12300 13422 137037 44642	9 9 7610 106	$^3A_2g \rightarrow {}^3T_2g$ $^3A_2g \rightarrow {}^3T_1g$ $\pi \rightarrow \pi *$,Intra-Ligand $\pi \rightarrow \pi *$	Octa-headral
6.	$C_{20}H_{36}Co_2N_8O_4Cl_4$	890 267 216	0.005 2.241 2.239	11235 37453 46296	50 2241 2239	$^4T_1g \rightarrow {}^4A_2g$ $\pi \rightarrow \pi *$ $\pi \rightarrow \pi *$	Octa-headral
7.	$C_{20}H_{36}Ni_2N_8O_4Cl_4$	882 772 719 542 370 283	0.003 0.003 0.001 0.002 0.058 1.927	11274 12853 14409 18450 26525 35714	30 30 10 20 580 1927	$^3A_2g \rightarrow {}^3T_2g$ $^3A_2g \rightarrow {}^3T_1g_f$ $^3A_2g \rightarrow {}^3T_1g_p$ $^3A_2g \rightarrow {}^3T_1g$ M →LCT, $n \rightarrow \pi *$ $\pi \rightarrow \pi *$	Octa-headral
8.	$C_{20}H_{36}Cu_2N_8O_4Cl_4$	881 542 291 209	0.031 0.003 1.464 0.330	11274 18450 34364 47846	31 30 1464 330	$^2Eg \rightarrow {}^2T_2g$ $^2E_2g \rightarrow {}^2T_1g$ $n \rightarrow \pi *$ $\pi \rightarrow \pi *$	Octa-headral
9.	$C_{20}H_{36}Zn_2N_8O_4Cl_4$	364 279	0.128 2.592	1280 25920	1280 25920	Intra-Ligand Intra-Ligand	Octa-headral
10.	$C_{20}H_{36}Cd_2N_8O_4Cl_4$	542 497 416 289 225	0.010 0.010 0.006 0.053 0.425	18450 20120 24038 34602 44444	100 100 60 530 4250	Intra-Ligand Intra-Ligand $n \rightarrow \pi *$ $\pi \rightarrow \pi *$	Octa-headral

Table (4) Shows the shapes of the UV Spectrum of complexes

 $C_{20}H_{48}Cr_2N_8O_{10}Cl_6$	 $C_{20}H_{40}Mn_2N_8O_6Cl_6$	 $C_{20}H_{48}Fe_2N_8O_{10}Cl_6$	 $C_{20}H_{36}Co_2N_8O_4Cl_4$
 $C_{20}H_{36}Ni_2N_8O_4Cl_4$	 $C_{20}H_{36}Cu_2N_8O_4Cl_4$	 $C_{20}H_{36}Zn_2N_8O_4Cl_4$	 $C_{20}H_{36}Cd_2N_8O_4Cl_4$

5. Elemental Analysis

The results of these analyses were listed in Table (5) and the values obtained in practice were compared with those calculated theoretically, showing clearly the degree of convergence between them. This proves the validity of the elemental ratios in the complexes. [29-32]

Table (5): Analysis of the elements of complexes.

Compound		C%	H%	N%	Cl%	M%
$C_{20}H_{48}Cr_2N_8O_{10}Cl_6$	Practical	29.21	3.84	14.86	24.67	13.54
	Theory	29.92	4.48	13.96	23.14	12.71
$C_{20}H_{40}Mn_2N_8O_6Cl_4$	Practical	32.49	3.72	16.05	19.73	23.09
	Theory	31.37	4.70	14.65	18.30	22.61
$C_{20}H_{48}Fe_2N_8O_{10}Cl_6$	Practical	30.05	4.06	14.88	24.94	14.27
	Theory	29.62	4.44	13.82	23.98	13.58
$C_{20}H_{36}Co_2N_8O_4Cl_4$	Practical	34.69	3.92	16.91	19.15	16.91
	Theory	33.85	5.07	15.79	19.74	16.50
$C_{20}H_{36}Ni_2N_8O_4Cl_4$	Practical	34.77	5.49	16.67	20.88	16.67

	Theory	33.89	5.08	15.81	19.77	16.38
$C_{20}H_{36}Cu_2N_8O_4Cl_4$	Practical	32.68	3.98	16.78	18.78	18.29
	Theory	33.42	5.01	15.59	19.48	17.54
$C_{20}H_{36}Zn_2N_8O_4Cl_4$	Practical	34.28	3.68	16.67	18.37	18.79
	Theory	33.24	4.98	15.51	19.39	18.00
$C_{20}H_{36}Cd_2N_8O_4Cl_4$	Practical	30.33	4.62	14.81	18.22	28.34
	Theory	29.41	4.41	13.72	17.15	27.45

6. Magnetic Sensitivity:

In addition to being used to determine the value of the correction factor that corrects the magnetic susceptibility readings, magnetic measurements have been employed in the diagnosis and investigation of transition metal complexes.[32-35]. From the results in Table (6), all measured complexes were Octahedral in shape

Table (6) Results of Magnetic measurements of complexes

NO	Complexes	Magnetic moment is effective Practically (B.M μeff)	Expected shape	Hybridizatio
1.	$C_{20}H_{48}Cr_2N_8O_{10}Cl_6$	2.48	Octahedral	SP^3d^2
2.	$C_{20}H_{40}Mn_2N_8O_6Cl_4$	8.154	Octahedral	SP^3d^2
3.	$C_{20}H_{48}Fe_2N_8O_{10}Cl_6$	5.56	Octahedral	SP^3d^2
4.	$C_{20}H_{36}Co_2N_8O_4Cl_4$	4.95	Octahedral	SP^3d^2
5.	$C_{20}H_{36}Ni_2N_8O_4Cl_4$	3.18	Octahedral	SP^3d^2
6.	$C_{20}H_{36}Cu_2N_8O_4Cl_4$	1.92	Octahedral	SP^3d^2
7.	$C_{20}H_{36}Zn_2N_8O_4Cl_4$	Diamagnetic	Octahedral	SP^3d^2
8.	$C_{20}H_{36}Cd_2N_8O_4Cl_4$	Diamagnetic	Octahedral	SP^3d^2

7. Molar Conductivity Measurement

All non-electrolytic complexes, with the exception of the Fe and Cr complexes, were determined to be electrolytic based on the synthesized complexes' observed electrical conductivity values. [36- 38]. As shown in Table (7).

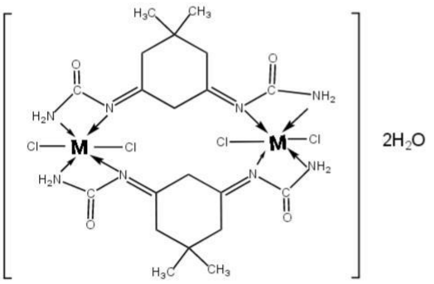
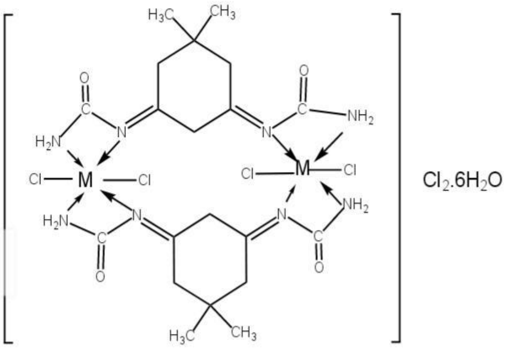
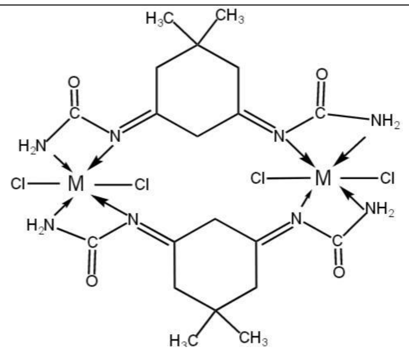
Table (7) Molar Electrical Conductivity values for complexes

No	Compounds	Λ_m $(S.cm^2.Mol^{-1})$
1.	$C_{20}H_{48}Cr_2N_8O_{10}Cl_6$	42
2.	$C_{20}H_{40}Mn_2N_8O_6Cl_4$	12
3.	$C_{20}H_{48}Fe_2N_8O_{10}Cl_6$	46
4.	$C_{20}H_{36}Co_2N_8O_4Cl_4$	6
5.	$C_{20}H_{36}Ni_2N_8O_4Cl_4$	9
6.	$C_{20}H_{36}Cu_2N_8O_4Cl_4$	11
7.	$C_{20}H_{36}Zn_2N_8O_4Cl_4$	21
8.	$C_{20}H_{36}Cd_2N_8O_4Cl_4$	19

8. Proposed shapes of the complexes

The shapes of the nodes were suggested by the results obtained.

Table (8) Proposed shapes of the complexes

 **M(II)=Mn**	 **M(III)=Fe, Cr**	 **M(II) =Co, Zn, Cu, Cd, Ni**

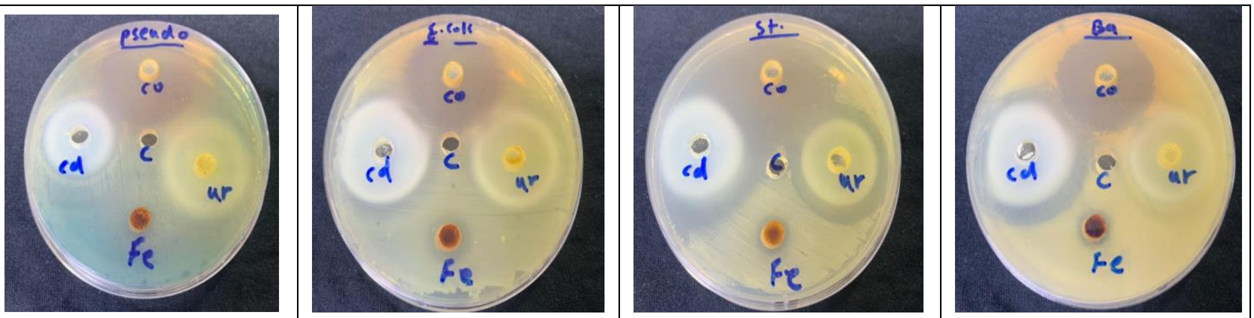
9. Antimicrobial Study of Metal Complexes.

In this study, four different types of bacteria were employed; two of the bacteria were found to be positively sensitive to the chromium dye (***Staphylococcus aureus***), ***Bacillus*** inhibition, and two of the other bacteria were found to be negatively sensitive (***E.coli* inhibition**). An in vitro antimicrobial test was conducted for Zinc Cobalt metal complexes. [39,40,41] The following was concluded:

Although ***bacillus*** and ***staphylococcus*** bacteria are resistant to antibiotics due to genetic factors as well, the prepared Zinc and Cobalt complexes demonstrated clear inhibition against gram-negative and gram-positive bacteria of four species. The effectiveness of these compounds' solutions is attributed to their ability to dissolve the outer cell wall, which causes the cell to exude fluids and ultimately die. As a result, compounds that affect the cell wall are among the most selective agents with a very effective therapeutic index. Owing to the structural differences between the bacterial and host cell walls [42].

Table (9) Inhibition values of the prepared complexes against Gram-negative and Gram-positive bacteria

No	Sample	Mg\ml	Gram-posative		Gramnegative	
			Staphy. zone(cm)	Bacillus zone(cm)	Spuedo. zone(cm)	E.coli zone(cm)
	$C_{20}H_{36}Zn_2N_8O_4Cl_4$	1	3.6	3.2	3	3.2
	$C_{20}H_{36}Co_2N_8O_4Cl_4$	0.25	5	3.4	3.5	3.5



$[Co_2(L_1)_2Cl_4]$ $[Zn_2(L_1)_2Cl_4]$ | $[Co_2(L_1)_2Cl_4]$ $[Zn_2(L_1)_2Cl_4]$ | $[Co_2(L_1)_2Cl_4]$ $[Zn_2(L_1)_2Cl_4]$ | $[Co_2(L_1)_2Cl_4]$ $[Zn_2(L_1)_2Cl_4]$

Figure (1) Inhibition zones of complexes against bacteria (*Bacillus, Spuedo, E.coli, Staphylococcus*)

9. Antioxidant activity of complexes

Gallic acid was used as a standard compound and neutralized with complexes: Cr(III), Mn(II), Fe(III), Co(II), Ni(II), Cu(II), Zn(II), Cd(II)DPPH is used to estimate reactive Oxygen species, and it then interacts with the complexes. Because of the transfer of Hydrogen from the complex to the DPPH molecule, this combination helps to change the colour of DPPH from Purple to Yellow. The color change was detected by UV Spectrophotometry (517) nm as shown in the figure and the following scheme to act as an inhibitor of Galic Acid and its complexes towards reactive oxygen species (ROS): GA < CoL1 < CuL1 < CdL1 < ZnL1 < MnL1 MnL1 and ZnL1 show more antioxidant activity [43-46].

Table (10) : Antioxidant activity values for complexes

Compound	Concentration	PI%	RSA%	IC_{50}

				mg/Ml
Gallic acid	0.008 0.004 0.002 0.001 0.0005	11.58 44.82 63.19 70.58 76.06	88.42 55.18 36.81 29.42 23.94	0.008
$[Co_2(L_1)_2Cl_4]$	0.056 0.028 0.014 0.007	19.13 38.18 55.25 60.92	80.87 61.82 44.75 39.08	0.027
$[Cu_2(L_1)_2Cl_4]$	0.062 0.031 0.016 0.008	28.05 40.43 49.48 58.12	71.95 59.57 50.52 41.88	0.028
$[Cd_2(L_1)_2Cl_4]$	0.060 0.030 0.015 0.007	29.46 49.43 48.03 57.18	70.54 50.57 51.97 42.82	0.041
$[Zn_2(L_1)_2Cl_4]$	0.049 0.024 0.012 0.006	27.46 49.03 55.33 59.78	72.54 50.97 44.67 40.22	0.055
$[Mn_2(L_1)_2Cl_4]2H_2O$	0.040 0.020 0.010 0.005	25.15 39.43 47.29 65.56	74.85 60.57 52.71 34.44	0.066
$[Fe_2(L_1)_2Cl_4]Cl_2.6H_2O$	0.064 0.032 0.016 0.008	22.15 38.43 44.29 62.56	77.85 61.57 55.71 37.44	0.075

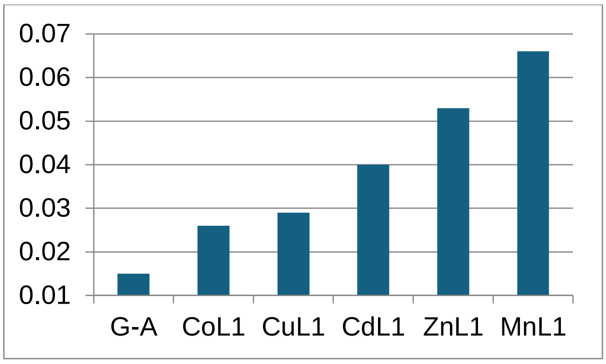


Chart (1) shows the difference between the IC50
values of the complexes

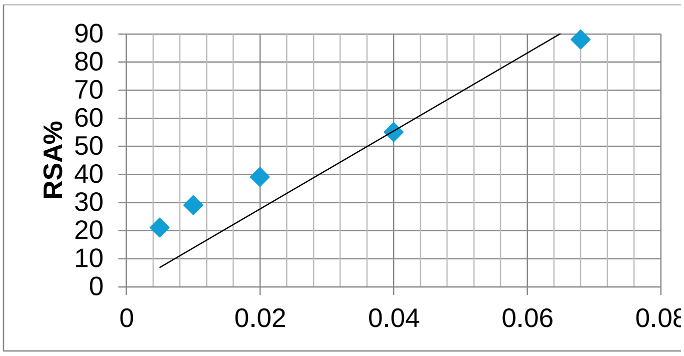
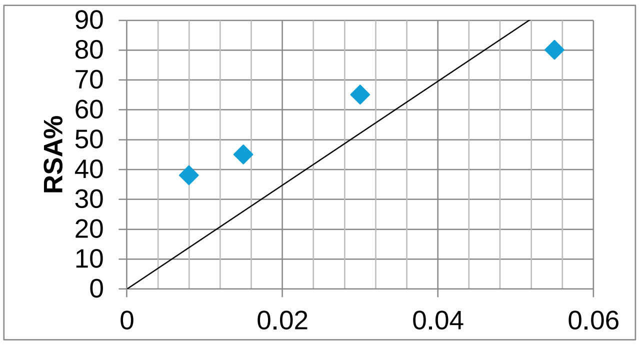
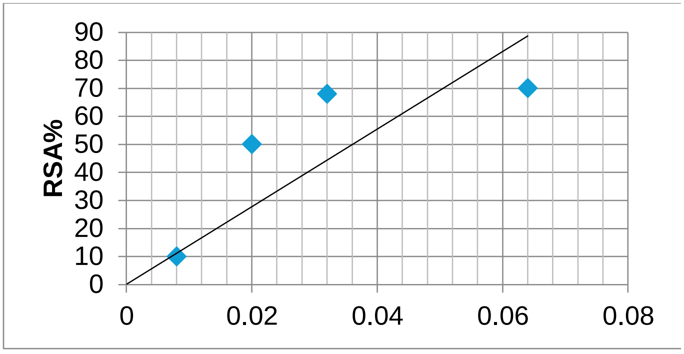


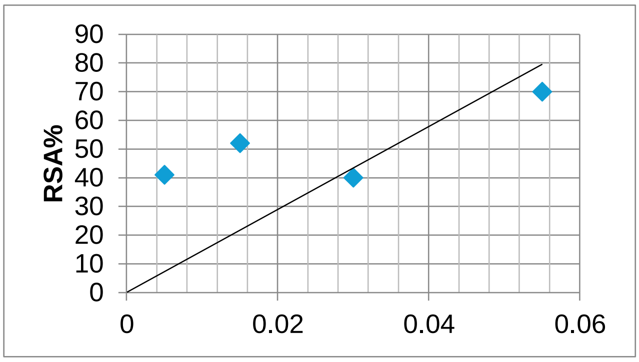
Chart (2) Galic Acide Inactivation Activity



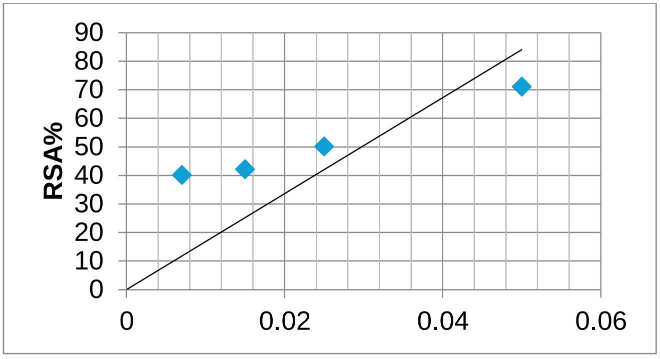
Scheme (3) Inhibition efficacy of the complex [Co2(L1)2Cl4]



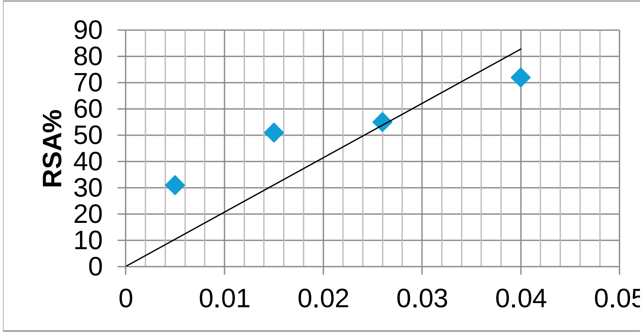
Scheme (4) Inhibition efficacy of the complex
[Cu2(L1)2Cl4]



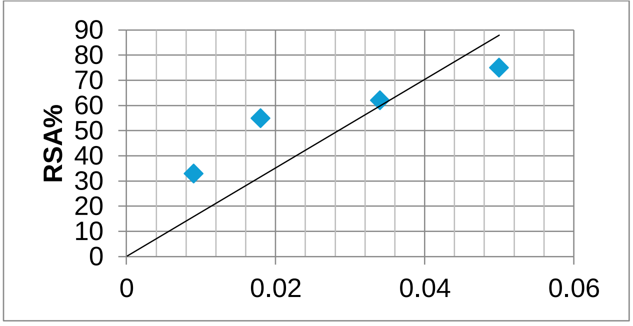
Scheme (5) Inhibition efficacy of the complex [Cd2(L1)2Cl4]



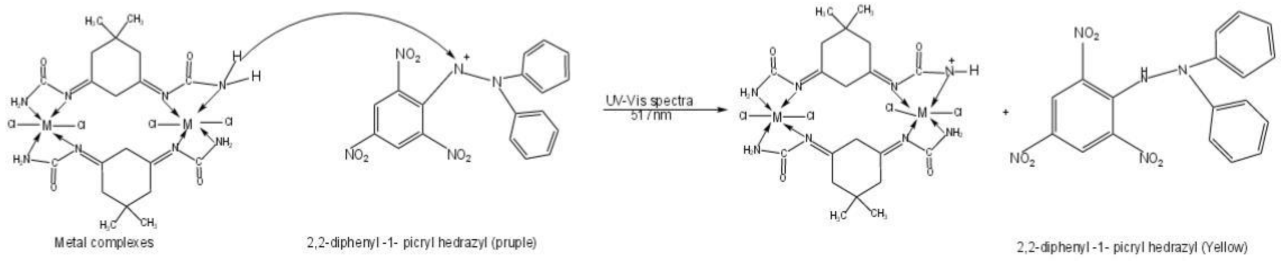
Scheme (6) Inhibition efficacy of the complex [Zn2(L1)2Cl4]



Scheme (7) Inhibition efficacy of the complex [Mn2(L1)2Cl4]



Scheme (8) Inhibition efficacy of the complex [Fe2(L1)2Cl4]



Scheme (9) shows the conversion of purple DPPH to the corresponding hydrazine (yellow) by adding a metal complex to DPPH due to proton transfer

Conclusion

The molecular structure of each prepared complex as well as the molecular structures of the prepared ligands was confirmed by magnetic susceptibility and electrical conductivity measurements. The synthesized complexes exhibited antioxidant and antibacterial properties. The synthesized complexes had different C=N absorption bands from their position in the ligand, which is evidence that the reaction took place. These new bands in the infrared spectrum validated the procedure used to produce the complexes. The complexes showed distinct (M-N) absorption bands, indicating that the electron pair of the nitrogen atom is what forms the bond between the metal ion and the ligand. -UV spectra also revealed the d-d absorption section of the complexes, which helped determine the final form of the complex. The octahedral electron transitions observed in the compounds.

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