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Spectral Studies of Mixed-Ligands Containing Lysine and 1,10-Phenanthroline

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Abstract

The synthesis and identification of the general composition mixed ligand complexes of M(II) ions [M(Lyn)2(phen)] are included in the research, where 1,10-phenanthroline ($C_{12}H_8N_2$), often abbreviated as "phen," functions as a secondary ligand and L-lysine ($C_6H_{14}N_2O_2$), often abbreviated as "LynH," as a primary ligand. At room temperature, the metal chlorides and their ligands were introduced to a reaction with ethanol serving as the solvent. The following molar ratio [(1:1:2) (metal): phen:2 Lyn -] was needed for the reaction. These included M = Mn(II), Cu(II), Ni(II), Co(II), Fe(II), and Cd(II) with M(II) ions. Additionally, we are investigating the bio-activity of several complexes that have been created against harmful bacteria, including *Bacillus (-), Escherichia coli (-), Staphylococcus (-), Pseudomonas (-)*, and *Staphylococcus S.P. (+).*

Keywords: Spectral analysis; L-Lysine; 1,10-Phenanthroline; Mixed ligands; Bioactivity

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1. Introduction

1,10-Phenanthroline (also called o-phenanthroline) is a heterocyclic organic compound used as an oxidation-reduction indicator due to characteristic color changes; turning faint blue when oxidized. It is used as an indicator to determine iron. [1–2]. The 1,10-phenanthroline (Figure 1) is very good nitrogen donor and generally acts as bidentate ligand in coordination chemistry, commonly abbreviated "phen," it forms strong complexes with most metal ions. In terms of its coordination properties, phen is similar to 2,2'-bipyridine. [3–5]

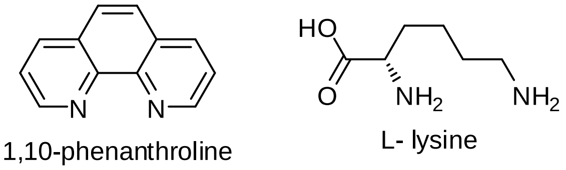


Fig. (1) 1,10-Phenanthroline and L-lysine

Metal complexes containing diimine ligands such as bipyridine and 1,10-phenanthroline have gained importance because of their versatile roles as building blocks for the synthesis of metallo-dendrimers and as molecular scaffolding for supramolecular assemblies, and in analytical chemistry, catalysis, electrochemistry, ring-opening metathesis polymerization and biochemistry [1–10]. Mixed ligand complexes can be a synthetic challenge to tune the properties of the transition metal complexes. [11] The complex of iron(II)tris(3-Br-phen) (3-Br-phen;3-bromo-1,10-phenanthroline) was prepared as a precursor of electro polymerization and the crystal structure of $[Fe(3\text{-}Br\text{-}phen)_3](PF_6)_2 \cdot CH_3CN$ with a distorted octahedral geometry has been investigated. [12]

Prominent among the various metal complexes employed so far in studies with DNA are those metallo–intercalators which incorporate either 1,10-phenanthroline (phen) or a modified phenanthroline moiety as a ligand. [13]Although DNA interactions of a number of $[M(phen)_2(LL)]^{n+}$ type complexes have previously appeared in the literature, relatively less attention seems to have been paid to systematic investigations inquiring into the effects brought about by changing M and LL in such complexes. [13–14]. L- lysine (Figure 1) is one of the twenty major amino acids and is considered an essential amino acid.[15] During the recent years , there has been significant interest in the coordination chemistry , the structural properties and the reactivity of metal complexes of amino acids [3,4,16].

In this paper we present the synthesis and study of Cu(II), Ni(II), Co(II), Mn(II), Fe(II) and Cd(II) complexes with amino acid L-lysine as a primary ligand and 1,10-phenanthroline as a secondary ligand have been used, respectively.

2. Experimental Part

All chemicals used were of reagent grade (supplied by either Merck or Fluka) and used as supplied. All the metal ions Mn(II), Co(II),Ni(II),Cu(II),Fe(II) and Cd(II) were of Analar grade (BDH). They were used in the form of chlorides without further purification.

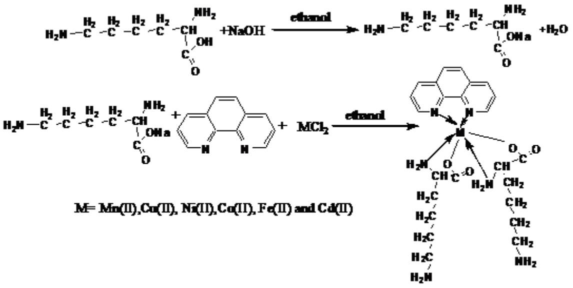
Instruments: FTIR spectra were recorded as K Br discs using Fourier transform Infrared Spectrophotometer Shimadzu 24 FTI.R 8400s. Electronic spectra of the prepared complexes were measured in the region (200- 1100) nm for 10^{-3} M solutions in ethanol at 25ºC using shimadzu-U.V-160.A Ultra Violet Visible- Spectrophotometer with 1.000 ± 0.001 cm matched quartz cell. While metal contents of the complexes were determined by Atomic Absorption(A.A)Technique using Japan A.A-67G Shimadzu. Electrical conductivity measurements of the complexes were recorded at room temperature for 10^{-3} M solutions of the samples in DMF using pw9527 Digital conductivity meter (Philips). Melting points were recorded by using Stuart melting point apparatus. chloride ion content were also evolution by (Mohr method),Magnetic susceptibility measurements were measured using Bruker magnet BM6 instrument at 298°K following the Faraday's method. The proposed molecular structure of the complexes were determinated by using chem. office program, 3DX (2006).

General synthesis

(1) 1,10-phenanthroline solution :prepared by dissolve [0.224 gm, 1m.mol] in ethanol (10ml).

(2) Sodium lycinate (Na^{+} lyn^{-}) **:** L- lysine [0.292gm, 2 mmol] was dissolved in 10 ml ethanol and added to 10 ml of ethnolic solution containing [0.08 gm (2mmol) of the sodium hydroxide , the solution was deprotonated according to the following reaction (scheme -2)

(3**)** Synthesis of complexes: An aqueous solution of the metal salt was added to the solution of the ligands in ethanol respectively using stoichiometric amounts (1:2:1) (metal: ligand: ligand)(M:2 Na^{+} lyn^{-} : phen) molar ratio, the mixture was stirred for half an hour at room temperature, crystalline precipitates observed. The resulting precipitates were filtered off , recrystallized from ethanol and dried at room temperature according to the following reaction (scheme1):



Scheme (1) Schematic representation of synthesis of the [M($C_{24}H_{34}N_6O_4$)] complexes

(4) Preparation of Microorganism suspension (16)

A) The micro- organism suspension was prepared by taking 2–4 colonies from all the studied micro-organism. Then it was inserted in the physiological solution in 0.85% concentration and was compared with Macferr land tube number 0.5 which is equal to 1.5×108 cell/mm. It is used for Petri dish preparation for the examination of biological activity against the under studied chemical compound.

B) Inhibition Activity Selection for the complexes in studied Micro-organism

The agar well diffusion method was used to see the effect of under studied chemical complexes on the micro-organism growth. This is done by using 20–25 ml from Nutrient agar medium for each Petri dish. The dish was incubated in incubator for 24 hours at (37°C) to make sure that no contamination would occur in the dish.

The dish was wetted in 10 milliliters of micro-organism which was prepared as mentioned in the previous paragraph which include 1.5×10^{8} cell/mm. Distributed evenly on the Nutrient Agar medium surface by using spreader. Bore was made on the cultured medium surface by using cork borer. The chemical complexes were made as 100 m ml per bore and left the central bore containing only DMF. The dishes were left for 1/2 hour in refrigerator at 4°C[16]. The dishes were incubated at (37°C) for 24 hours. The biological activity for the complexes was defined by measuring the diameter of the inhibition area surrounding each bore in millimeters.

3. Results and Discussion

Products were found to be solid crystalline complexes, which have been characterized through the following: Solubility, Molar conductivity, Determination the percentage of the metal in the complexes by (AAS), Spectroscopic Method [FTIR and UV-Vis]. Table (1) shows the physical data for the prepared complexes which show different melting points, all of them were higher than the two ligands, All the complexes are colored, non-hydroscopic and thermally stable solids (Table 1), indicating a strong metal-ligand bond.

Table (1) Physical properties of the compounds

Compounds	MW	Color	M .p (de) °C
LynH ($C_6H_{14}N_2O_2$)	146.19	White	> 350
phen($C_{12}H_8N_2$)	180.12	White	114-117
Mn ($C_{24}H_{34}N_6O_4$)		Yellow	300 ^{d} >
Co($C_{24}H_{34}N_6O_4$)	529.50	Brown	300 ^{d} >
Ni ($C_{24}H_{34}N_6O_4$)	529.26	Green	300 ^{d} >
Fe ($C_{24}H_{34}N_6O_4$)	526.42	brown-red	300 ^{d} >
Cu ($C_{24}H_{34}N_6O_4$)	531.11	Blue	300 ^{d} >
Cd($C_{24}H_{34}N_6O_4$)	582.98	White	300 ^{d} >

Compounds	Λm Ω^{-1} cm^2 mol^{-1}	Cl%	%Metal Theory	% Metal Exp.
LynH ($C_6H_{14}N_2O_2$)	1.0	-	-	-
phen($C_{12}H_8N_2$)	2.6	-	--	
Mn ($C_{24}H_{34}N_6O_4$)	5.69	Nill	11.03	12.4
Co($C_{24}H_{34}N_6O_4$)	7.68	Nill	11.13	12.4
Ni ($C_{24}H_{34}N_6O_4$)	9.79	Nill	11.09	11.61
Fe ($C_{24}H_{34}N_6O_4$)	8.28	Nill	10.61	11.4
Cu ($C_{24}H_{34}N_6O_4$)	8.22	Nill	11.90	11.4
Cd($C_{24}H_{34}N_6O_4$)	10.59	Nill	19.28	------

d: Decomposition points of the corresponding molecule

1,10–Phenanthroline is soluble in alcohol and acetone and hardly soluble in water. The complexes are insoluble in water but soluble in common organic solvents such as ethanol, methanol, ethyl alcohol, acetone, chloroform, DMF and DMSO.

The atomic absorption measurements and chloride ion content (Table 1) for all complexes gave approximated values for theoretical values. In conclusion, our investigation this suggest that the ligands acid L-lysine and 1,10-phenanthroline coordinate with M (II) forming octahedral geometry (Fig. 3).

The molar conductance of the complexes value range between (5.69-10.59 ohm^{-1} cm^2 mol^{-1}) of the complexes which was carried out in ethanol solvent indicates that the complexes under study are non-electrolytic in nature. The electrical conductivity of these complexes found in the order:
Cd > Ni> Fe > Cu > Co > Mn.

Insolubility of these complexes in water and their non-electrolytic nature provide sufficient evidence for covalence of the ligands [18].

The most important infrared spectral bands that provide conclusive structural evidence for the coordination of the ligands to the central metal ions. The assignment of some of the most characteristic FTIR bands of the complexes are shown in Table (2) together with those of two ligands recorded for comparative purposes and facilitate the spectral analysis. The IR spectra of the all complexes show characteristic vibrations of the aromatic nucleus [ʋ (C=C), ʋ (C=N)] of 1,2-phenanthroline near 1590, 1503 and 1430 cm^{-1} respectively, while (C-H) deformation vibrations are about 726 cm$_{-1}$ (1,2-phenanthroline) [19]. Regarding the amino acids as secondary ligand, distinct bands appearing in the 1545-1570 cm^{-1} and 1420-1430 cm^{-1} regions in the IR spectra of the complexes, which are attributable to the asymmetric and symmetric stretching vibration of the carboxylate group ʋ (COO$^-$). These bands are shown, however in the 1560- 1570 cm^{-1} and 1388-1392 cm^{-1} regions for complexes. [20-21]. The separation ʋ $_{\text{asym}}$- ʋ $_{\text{sym}}$ values (Table 2) are taken as an evidence for the participation of the carboxylate group, ʋ (N-H) band appears in the 3290-3330 cm^{-1} region, hence it can be concluded that the nitrogen of the amino group is involved in coordination. [22-23].It is found from the IR spectra of the complexes that there are wide and strong band at 632-648 cm^{-1} for ʋ(M-N) bonding and 450-516 cm^{-1} for ʋ (M-O) which are assigned to metal stretching vibration [20-24].

The magnetic moment data are presented in Table (3). The Mn(II) complex showed a value of 5.38 μB, which is slightly lower than the spin only value of 5.92 μB for high spin octahedral Mn(II) complexes. [23]. The magnetic moment of the Fe(II) complex was 5.78 μB, which supports its high spin octahedral geometry. [25]. The observed magnetic value of Ni(II) complex was 3.18 μB, which is in good agreement with a spin free octahedral geometry. [24] The Cu(II) complex exhibited a value of 1.83 μB, which suggests an octahedral geometry around the central metal ion. Cd(II) complex is diamagnetic in nature and its electronic spectra do not furnish any characteristic d-d transitions except charge transfer (LMCT) bands as expected for d^{10} systems. The electronic spectra of the ligands and there complexes were recorded in DMF and their assignments are given in Table (3). The free ligand 1,10–Phenanthroline spectral data display two bands at 311 nm (32154 cm^{-1}), 338 nm (29586 cm^{-1})) attributed to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions and the spectrum of the free ligand (L-lysinee), figure (3) exhibits absorption peak at (280 nm) (35714.28 cm^{-1}) and an intense peak at 320 nm (31250.0 cm^{-1}), which assigned to ($\pi \rightarrow \pi^*$), and ($n \rightarrow \pi^*$) transition respectively [16,17].

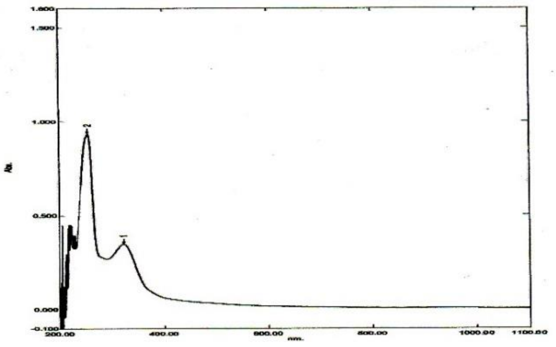


Fig. (2) Electronic spectrum of the ligand (L-lysine)

The electronic spectra of Co(II) complex recorded in DMF solution display three absorption bands at

(34482, 32154 and 17123) cm^{-1} corresponding to the following transitions, respectively: LMCT, LMCT, and $^4T_{1g(F)} \rightarrow {}^4A_{2g(F)}$. This shows that this complex has octahedral geometry.

The electronic spectra of Mn (II) complex exhibited three spin allowed bands in the region 668nm (14970; cm^{-1}, 536nm (18656 cm^{-1}) and 410 nm (24339 cm^{-1}) assigned to the transitions $6A_{1g} \rightarrow 4T_{1g}$ (4G) (υ1), $6A_{1g} \rightarrow 4T_{2g}$ (4G) (υ_2) and $6A_{1g} \rightarrow 4E_g$, $4T_{1g}$ (4P) (υ_3), respectively, indicating octahedral geometry [24-25].

The reflectance spectra of the Ni(II) complex, exhibit two strong bands at 662nm (15009 cm^{-1}) and 407 nm (24515 cm^{-1}) ,assignable to $3A_2g$ (F) → 3T1g (F) (υ_1) and $3A_{2g}$ (F) → $3T_{1g}$ (P) (υ_2) respectively. The (υ_2/υ_1 ratio for the chelate is 1.633 occurs in the usual range (1.600- 1.820) for octahedral Ni(II) chelates 26. The spectral bands are well within the range observed for hex coordinate octahedral complexes reported earlier [23-26].

The electronic spectra of the Co(II), complex show absorption bands at 311 nm (32154 cm^{-1}) , 290 nm (34482 cm^{-1}) and 584 nm (17123 cm^{-1}) due to the presence of a charge transfer (LMCT) and $4T1g(F) \rightarrow 4A2g(F)$ [26-28].

The electronic spectra of the Fe(II) complex exhibited three bands at 310nm (32258 cm^{-1}) 300 nm (33333 cm^{-1}) and 510nm (19607 cm^{-1}) due to the presence of a charge transfer (LMCT) and $^5T_{2g} \rightarrow {}^5E_{2g}$ (Fig. 3). The electronic spectra of the Cu(II) complex exhibited three bands at 352nm (28409 cm^{-1}) 249 nm (40160 cm^{-1}) and 590 nm (16949 cm^{-1}) due to the presence of a charge transfer (LMCT) and $2E_g \rightarrow 2T_{2g}$.

The metal complexes are of the high-spin type and paramagnetic, except for the Cd(II) complex, which is diamagnetic. The values of μeff suggest octahedral coordination around the metal ions. [25-27].

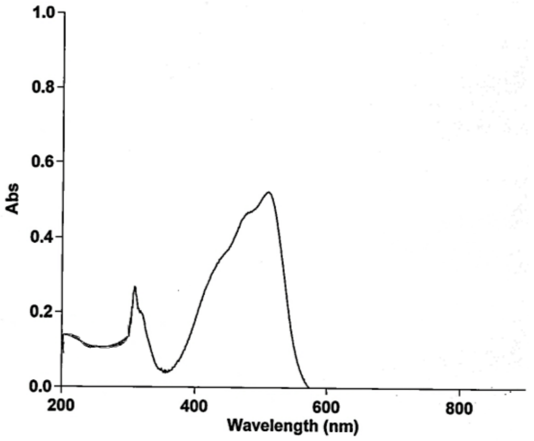


Fig. (3) The (UV-Vis) Spectra of Fe ($C_{24}H_{34}N_6O_4$)

The zone inhibition of bacterial growth was measured in mm depending upon the diameter as shown in Table (4) and Fig. (10).

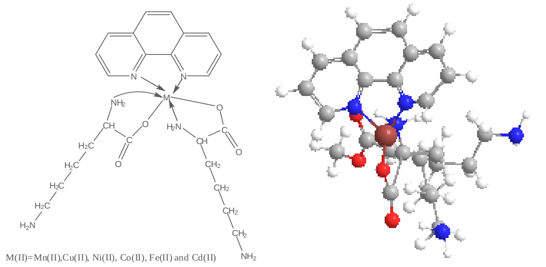
Table (4) Inhibition circle diameter in millimeter for the bacteria after 24 hrs incubation paid and 37°C for complexes

Compounds	*E .coli*	*Pseudomonas*	*Staphylococcus*	*Bacillus*
Control(DMF)	5	5	5	5
Mn ($C_{24}H_{34}N_6O_4$)	45	60	60	60
Ni ($C_{24}H_{34}N_6O_4$)	40	60	50	50
Fe ($C_{24}H_{34}N_6O_4$)	40	45	40	35
Cu ($C_{24}H_{34}N_6O_4$)	40	60	50	50
Cd($C_{24}H_{34}N_6O_4$)	45	65	50	40

On the basis of observed zones of inhibition, All the metal-mixed ligand complexes are active against all tested organisms *Escherichia coli(-), Pseudomonas(-), Bacillus(-) and Staphylococcus S.P* (+), which in fact is in agreement with the literature [28-29]. The antibacterial activity of the metal chelates was found to be in the order: Mn >Cd (II)> Ni (II) >Fe (II). Hence produce metal chelates can be employed as antibacterial.

Proposed molecular structure

Studying complexes on bases of the above analysis, the existence of Hexa coordinated [$M(C_6H_{13}N_2O_2)_2(C_{12}H_8N_2)$], were M(II)= Mn(II) , Cu(II), Ni(II), Co(II), Fe(II), and Cd(II). Proposed models of the species were built with chem. 3D shows in Fig. (4).



M(II)=Mn(II),Cu(II), Ni(II), Co(II), Fe(II) and Cd(II)

Fig. (3) The proposed structure and 3D-geometrical structure of the complexes

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Table (2) FTIR spectral data of the Ligands and there complexes

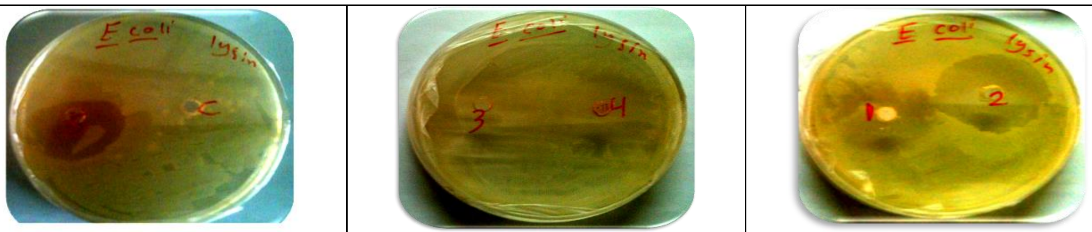
Compound	NH_{sym} str	$CH_{(py)}$ str	$(CH)_{cyclic}$	υ (C=N)	υ (C=C)	υ(C–O)	υ∂ (C-O)	$\upsilon(\text{-COO}^-)_{\text{asym sym)}}$	$\rho\upsilon\text{COO}^-)_{\text{asym sym)}}$	M-O	M-N
Lyn H ($C_6H_{14}N_2O_2$)	3240							1618 vs (1411s)	207		-
Phen ($C_{12}H_8N_2$)	3240- 3047br	-	-	1577	1508	-	-	-	-		-
Mn ($C_{24}H_{34}N_6O_4$)	3218s	2983vs	2923s	1589vs	1589 vs	1283s	457	1483s (1386s)	97	457	632s
Co($C_{24}H_{34}N_6O_4$)	3168w-	3136s 2854w	2974m	160w	1573vs	1283m	457m	1465s (1323s)	142	450m	640 m
Ni ($C_{24}H_{34}N_6O_4$)	3194br-s	3136m 2854w	2924w	1590s	1570s	1269m	505m	1465vs (1377vs)	88	450w	648m
Fe ($C_{24}H_{34}N_6O_4$)	3356 br s	3240 br-s	2989w	1620s	1520w	1284m	497m	1497vs (1373s)	124	503w	644m
Cu ($C_{24}H_{34}N_6O_4$)	3384 br	3058m	2925w	1581vs	1504m	1284m	588s	1469vs (1377vs)	92	516m	632m
Cd($C_{24}H_{34}N_6O_4$)	3315s	3085m	2927w	1577vs	1500 vs	1282s	583w	1467vs (1371vs)	98	503m	640m

Sym: symmetric, asy: asymmetric, am: amide, py: pyridine, o.p: out of plane, str: stretching, v.s: very strong, s: strong, m: medium, w: week, sh: shoulder

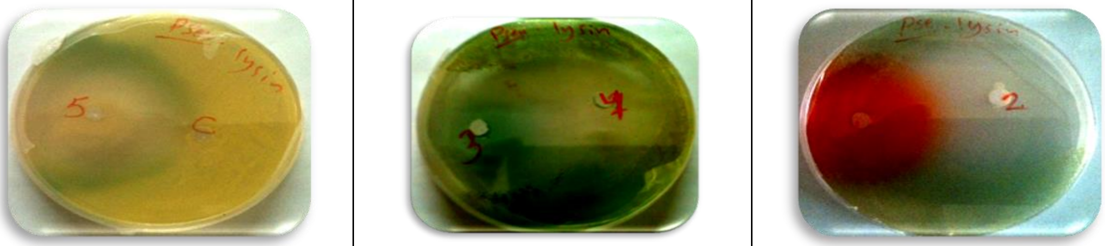
Table (3) Electronic Spectral data, magnetic moment, of the studied complexes and two ligands

Compounds	λ max(nm)	υ′(cm^{-1})	Assignments	μ_{eff} (BM)
Lyn H ($C_6H_{14}N_2O_2$)	280 320	35714 31250	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$	-
Phen ($C_{12}H_8N_2$)	311 338	32154 29586	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$	-
Mn ($C_{24}H_{34}N_6O_4$)	301 410 536 668	33222 14970 18656 14970	LMCT 6A 1g → 4Eg, $4T_{1g}$ (4P) $6A_{1g} \rightarrow 4T_{2g}$(4G) $6A_{1g} \rightarrow 4T_{1g}$ (4G)	58
Co($C_{24}H_{34}N_6O_4$)	290	34482	LMCT	4.90

	311 584	32154 17123	LMCT $4T_{1g}(F) \rightarrow 4A_{2g}(F)$.	
Ni ($C_{24}H_{34}N_6O_4$)	314 407 666	31847 24515 15009	LMCT $3A_{2g}$ (F) → $3T_{1g}$ (P) $3A_{2g}$ (F) → $3T_{1g}$ (F)	3.18
Fe ($C_{24}H_{34}N_6O_4$)	300 310 510	33333 32258 19607	LMCT LMCT $^5T_{2g} \rightarrow {}^5E_{2g}$	5.78
Cu ($C_{24}H_{34}N_6O_4$)	249 352 590	40160 28409 16949	LMCT LMCT $2Eg \rightarrow 2T_{2g}$	1.83
C d($C_{24}H_{34}N_6O_4$)	311	32154	LMCT	Diamag

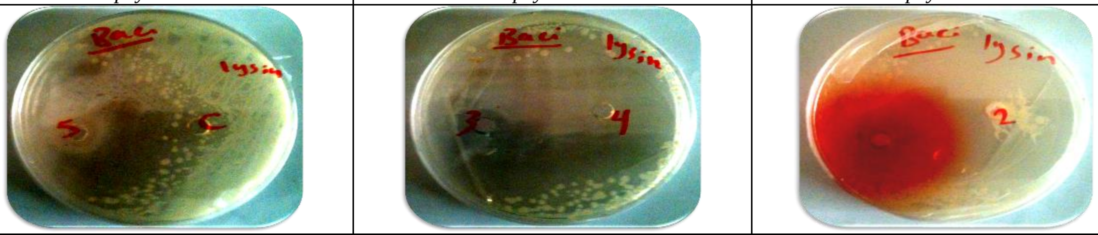


E .coli *E .coli* *E .coli*



Pseudomonas *Pseudomonas* *Pseudomonas*

Staphylococcus *Staphylococcus* *Staphylococcus*



Bacillus *Bacillus* *Bacillus*

5-Mn($C_{24}H_{34}N_6O_4$)

3-Cu ($C_{24}H_{34}N_6O_4$)
4-Ni($C_{24}H_{34}N_6O_4$)

1-Fe ($C_{24}H_{34}N_6O_4$)
2-Cd($C_{24}H_{34}N_6O_4$)

Fig. (6) The antimicrobial activity of complex 1, 2, 3, 4, 5 appear the inhibition zones against pathogenic bacteria (*E .coli, Pseudomonas, Staphylococcus and Bacillus*)