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Spectroscopic, Structural and Antibacterial Activity of Mixed Ligand Complexes from Schiff Base with Anthranilic Acid

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Abstract. Mixed ligand complexes included Schiff base derivative ligand (L1) have been produced by condensed {3-amino-1-phenyl-2-pyrazolin-5-one (APO)} with o-phthalaldehyde and anthranilic acid (L2) with some transition metal ions. Diagnosis of prepared ligand (L1) by ¹HNMR, mass spectroscopy, (CHN) elemental analysis and UV-Visible. The complexes were typified by molar conductance, UV-Visible, FTIR, atomic absorption, magnetic susceptibility and elemental analysis techniques. Octahedral geometry are suggested for the metal complexes based on the analytical studies. All these compounds were determined against with two type of bacteria; Gram positive and Gram negative.

Keywords

Schiff base, complexes, mixed ligand, analytical studies.

1. Introduction

The compounds of Schiff base (SB) are active azomethine group (-CH=N-). They are prepared by Hugo Schiff that frequently time distinguished as a result of yellow colored light [1]. In coordination chemistry and medicament, this type of ligands (Schiff base) stand for fundamental compounds as a consequence of the existence of (-CH=N-) with antiseptic performance, catalysis, anti-oxidative, antifungal, enzymatic reaction, bioinorganic prototyping and magnetism investigations. Pyrazole stands for the organic compound with the form (C₃H₃N₂H). It is weak base and has hetero electric designated through a 5-membered noose of 3(C) and 2- adjacent N atoms [2,3]. Protuberant drugs having pyrazole loop are Celebrex (silcoxib), and motivated steroids [4-7]. New pyrazol ring compounds have outstanding copolymerization and endure a rinse in biological solvents at what time employed for eyes and eye lens. In visible light region, they possess tremendous peak of absorbance and



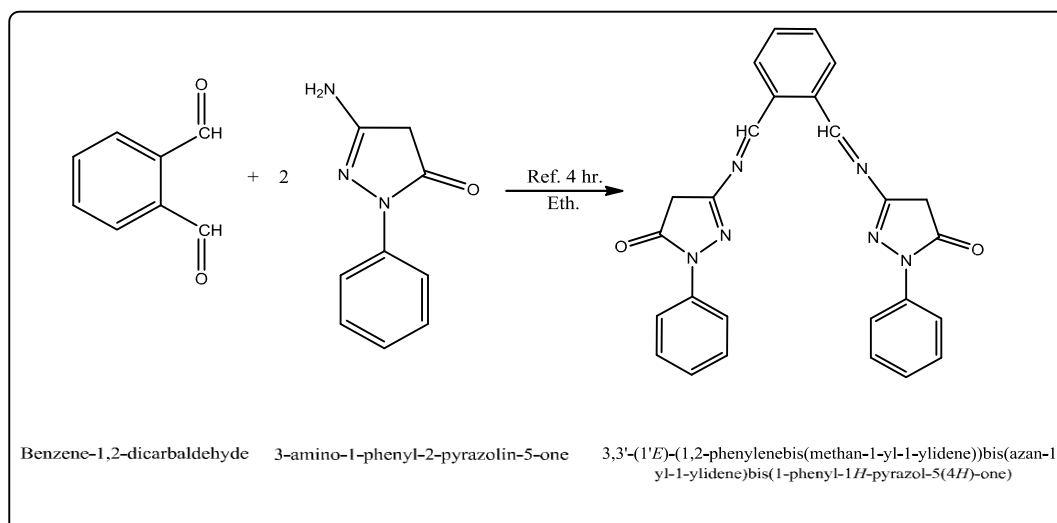
role as yellow color reactivity. A formulation of 5-amino-2,4-dihydro-2-phenyl-3H-pyrazole-3-one or (APO) is (C₉H₉N₃O) [8,9] and it is imperative in organic behaviors (10). So, the synthesis of newfangled derivatives ligands was involved the attention of scholars in all over the world. The synthesis of dual Spiro- compounds including unforeseen pyrazole via dual processes; APO condensation along with benzaldehyde and dimerization [11]. Ni and Cd complexes of new (SB) resulting from 5-Amino-2-phenyl-2,4-dihydro-pyrazol-3-one with 4-chlorobenzaldehyde (A) , 2-Hydroxy-benzaldehyde (B) and 4-Hydroxy- Benz aldehyde (C) have been prepared and categorized by molar conductivity measurements , elemental analysis, FTIR , UV- vis , ¹HNMR, mass spectrometer as well as magnetic susceptibility. In this research, we prepared and characterized new complexes with ligand Schiff base and with some meta(II) ions .

2. Materials and device :

All chemical used in laboratory work has purchased from distinguished sources with highest purity ready without further purity . Melting points were carried out via Stuart. Elemental micro analysis for the ligand was carried out by Euro (EA 3000) instrument. ¹H NMR spectrums are achieved via Bruker DRX system (400 MHz). Ultra Violet-Visible Spectrophotometer spectrums were performed on a Shimadzu UV- 160A in KBr discs on (4000-400) cm-1range.The IR-spectra have been verified by FTIR-8400S Spectrophotometer. Metal contents (A.A.S) of the complexes were determined by using atomic absorption method by means of AA 620G Shimadzu spectrophotometer. The Chloride substances of compounds have been specified by testing all complex and decomposed with nitric acid and diluted with water. Measured magnetic findings were performed by Bruker BM6 instrument at room temperature by using the Faraday's method.

2.1 Synthesis of ligand Schiff base

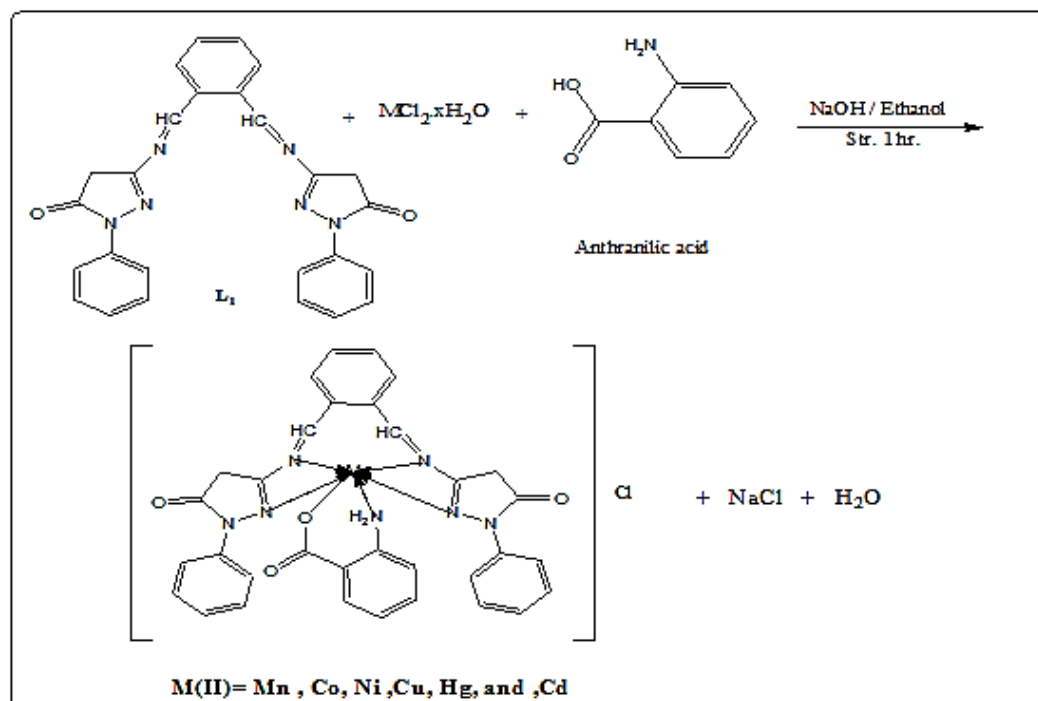
APO (0.3g, 2mol) in absolute ethanol (5ml) has inserted in the refluxing solution of (0.134g, 1mol) o-phthalaldehyde for the related solvent (10 ml) in (100 ml) round-bottomed flask with three or four drops of glacial acetic acid[11]. The mixture was excited in bath water by refluxing at 70 to 80° C for 4 hours with uninterrupted stirring. The solution color was altered from yellow to orange to brown. Then, the brown color was generated. The solid outcome was constituted as a result of filtration, cleaned by absolute ethanol and lastly dehydrated in open air. It cleansed via re-crystallization using absolute acetone and then at room temperature dehydrated. Scheme (1) explains a formation of ligand, yield: 80%, m. p.: 250° C.



Scheme (1): The Ligand (L₁) Synthesis

2.2. Preparation of mixed ligand complexes

The solution of L_2 (0.137g, 1mol) besides NaOH (0.04g, 1mol) and L_1 (0.448g, 1mol) in ethanol (5ml) has inserted to stirred metal chloride 1 mol as in Mn^{+2} (0.126g), Ni^{+2} (0.237g), Co^{+2} (0.237g), Cd^{+2} (0.201g), Cu^{+2} (0.17g) and Hg^{+2} (0.272g) liquefied in (5ml) ethanol. The resultant mixture was stirred for 60 minutes. The precipitant has filtered and dehydrated at room temperature for all day.



Scheme (2): Synthesis of $[M(L_1)(L_2)]Cl$ Complexes

2.3. Antibacterial activity

SB and their metal compounds have investigated with four type of bacteria as *psuedomonas aruginosa* (1), *Escharia coli* (2), *Staphylococcus aureus* (3) and *Streptococcus pyogenes* (4) by disc diffusion technique. The used solutions in the organic investigation are organized via dimethyl sulfoxide (DMSO) as solvent and they are provided as single concentration of 0.001M. The dishes are hatched at room temperature for a complete day. Inhibiting zones (IZ) in mm has been formed after a complete day and adopted as a standard for the synthetic chemical compound intensity consequence on the extension of refined specific bacteria strains.

3. Results and discussion:

All the complexes have been soluble in DMSO and DMF but insoluble in water. The CHN analysis and the sensible features of the compounds were listed in table-1. At room temperature, the molar conductivity magnitudes of (L_1) and (L_2) ligand have been determined in DMSO solvent in (0.001) M solution. Based on magnitudes of L complexes (31.22 - 37.48), complexes have been found to be electrolytic [12], and the ratio is (1:1).

The complexes can be symbolized as $[M(L_1)(L_2)]Cl$; where M=meta(II) ions; (L_1 =Schiff base ligand) and (L_2 = anthranilic acid). the code of the complexes were $[Mn(L_1)(L_2)]Cl$ was La Pale-brown, $[Co(L_1)(L_2)]Cl$ was Lb brown, $[Ni(L_1)(L_2)]Cl$ (Lc) Pale-brown, $[Cu(L_1)(L_2)]Cl$ (Ld) Dark brown, $[Cd(L_1)(L_2)]Cl$ (Le) Pale-brown and $[Hg(L_1)(L_2)]Cl$ (Lf) Dark brown.

Table 1. Physical Properties and Elemental Microanalysis Compounds

Code	M.wt	$^{\circ}C$	Theoretical (Calc.)					
			C	H	N	O	Cl	M
L1	448.16	250	69.63	4.49	18.74	7.14	--	---
			69.45	4.32	18.56	7.23		
La	675.00	124	58.72	3.88	14.53	9.48	5.25	8.14
			58.65	3.68	14.42	9.53		8.28
Lb	678.99	183	58.37	3.86	14.44	9.43	5.22	8.68
			58.22	3.67	14.32	9.33		8.34
Lc	678.75	273 dec.	58.39	3.86	14.45	9.43	5.22	8.65
			58.21	3.71	14.27	9.30		8.24
Ld	683.60	203	57.98	3.83	14.34	9.36	5.19	9.30
			57.78	3.69	14.22	9.65		9.13
Le	732.47	234	54.11	3.58	13.39	8.47	4.84	15.35
			54.35	3.73	13.16	8.38		15.14
Lf	820.65	284 dec.	48.30	3.19	11.95	7.80	4.32	42.44
			48.12	3.04	11.78	7.68		42.32

3.1. 1H -NMR spectrum of formed L_1 ligand in $DMSO-d_6$ has been presented in Figure (1). A singlet signal shown in $\delta = 2.53$ and 3.31 ppm is possibly assigned to $DMSO$ solvent [13] and (CH_2) of pyrazole (4H, s). The manifold signals under $\delta = 7.12-8.09$ ppm has been allocated for the aromatic protons (14H, m) of phenyl, and the singlet signal under ($\delta = 8.51$ ppm) has been in accordance with azomethine proton ($HC=N$) (2H, d) [11,14], table (2).

Table 2. 1H -NMR Data for L_1

Functional group	δ (ppm)
$DMSO-d_6$	2.53
Ar-H	7.12-8.09 (14H, m)
Py-H	3.31(4H, s)
N=C-H	8.51 (2H, s)

3.2. Mass spectrum: The foremost extreme resolution mass spectrometer of created L1 ligand has been depicted in figure (1). A molecular ion peak has been detected at $m/z = 445.2$ for ($C_{26}H_{20}N_6O_2$) that had been extremely reliable with the theoretic magnitude [11,13–15] as in table (3).

Table 3. Mass Details For L1

Fragment	Mass m/z
$[M^+] = [C_{26}H_{20}N_6O_2]^+$	445
$[C_{20}H_{15}N_5O_2]^+$	357
$[C_{19}H_{15}N_5O]^+$	330
$[C_{17}H_{13}N_3O]^+$	273
$[C_{10}H_8N_3O]^+$	175
$[C_7H_5N_2O]^+$	119
$[C_6H_5N_2]^+$	93
$[C_3H_3N]^+$	42

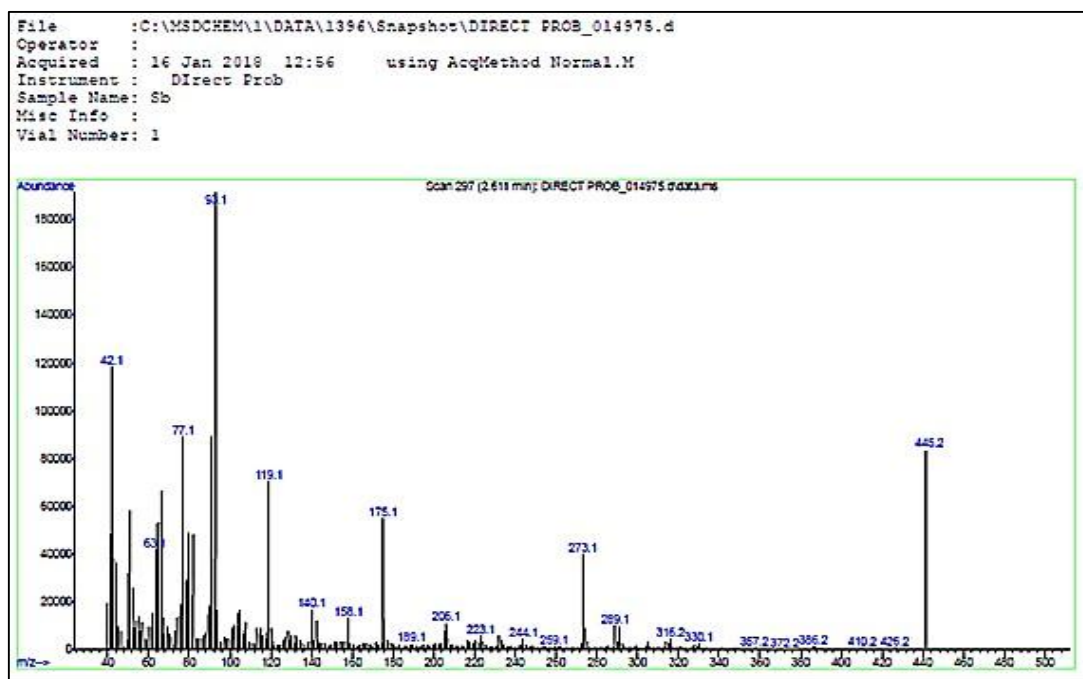


Figure (1): Mass Spectrum of (L1)

3.3. The FTIR spectra: of the free ligand was documented in the 4000 – 400 cm^{-1} area on KBr Pellets. IR band of L has shown the weak absorbance band at 3126 cm^{-1} that allocated for (C-H) aromatic stretching vibration. In 1699 cm^{-1} , band has been with (C=O ring pyrazole) stretching vibration. A resilient band in 1670 cm^{-1} has been with stretching vibration of (C=N). Lastly, at 1637 cm^{-1} , the band has been with stretching vibration of (C=C) as depicted in table (4). [16]. The FTIR spectrums of organized complexes have been detected in the fundamental absorbance bands with respect to (L1) spectrum. For Table (4), FTIR bands of ligand (L2) have bands of $\nu(\text{O-H})$, $\nu(\text{NH}_2)$, $\nu(\text{COO})$ asy and $\nu(\text{COO})$ sym. All complexes have absorption spectrum within (3479-3290) cm^{-1} range for $\nu(\text{N-H})_{\text{asy,sym}}$ stretching vibration for L2, that has been moved to upper frequency as compared with initial material spectrum. Accordingly, complexes ions have been associated with this group. A broad $\nu(\text{O-H})$ band has been vanished in six complexes. The bands of absorption for stretching vibration for imine cluster of pyrazole ring and (HC=N) Schiff base were in complex spectrums at the supreme frequency (1674-1681) cm^{-1} in comparison of free ligand spectra in (1670) cm^{-1} . Under conditions of lower frequency (1615-1625) cm^{-1} as compared with original spectrum at (1637) cm^{-1} . This spectrum location shifting has steadfast indication of coordinating amid nitrogen atom in this group along with the metal ion [17–19]. Bands of absorption ν_{asy} along with $\nu_{\text{sym}}(\text{COO})$ in the complexes have been moved to upper frequencies at (1535-1562) cm^{-1} for $\nu(\text{COO})_{\text{asy}}$ and minor frequencies for $\nu(\text{COO})_{\text{sym}}$ at (1307-1323) cm^{-1} . Consequently, a change amid $\Delta_{\text{asy-sym}}$ has been equivalent to (212-255) cm^{-1} . Carboxylate ion coordinating with metal ions has been as a monodentate donor [20–22]. IR spectrums concerning complexes have presented newfangled absorption bands which demonstrated the ligand coordination at (462-497) cm^{-1} with fundamental metal ion throughout nitrogen atom of imine group of SB. In (412-470) cm^{-1} , the amine group of Anthranilic acid are feasibly stated as $\nu(\text{M-N})$. Also, the detected spectrums with absorption band were at (563-570) cm^{-1} because of $\nu(\text{M-O})$ [23,24]. The bands of absorption of organized complexes that are between (1694-1999) cm^{-1} . They have been attributable to $\nu(\text{C=O})$ of pyrazole ring. Under conditions of (3060-3151) cm^{-1} in complexes, bands of absorption have been belonged to $\nu(\text{C-H})$ aromatic [16]. .

Table 4. FTIR Band Details For The Ligand And Their Complexes

	ν (O-H)	ν (N-H) _{asy} &sym	ν (C-H) _{Arom.}	ν (C=O)	ν (C=N) _{py}	ν (C=N) _{Sch.}	ν (COO)	Δ (asy-sym)	ν (M-O)	ν (M-N) _{Sch.} ν (M-N) _{anth.}
L1	----	----	3126	1699	1670	1637	----	----	----	----
L2	3390	3321- 3210	2586	1716	----	----	----	----	----	----
LA	----	3475- 3417	3151	1699	1678	1620	1546- 1319	227	567	497- 470
LB	----	3479- 3414	3140	1695	1681	1625	1543- 1323	220	563	466- 439
LC	----	3305- 3290	3136	1697	1681	1616	1546- 1319	227	567	497- 466
LD	----	3479- 3450	3062	1694	1681	1616	1535- 1323	212	567	497- 465
LE	----	3475- 3367	3062	1695	1674	1615	1540- 1307	233	563	462- 412
LF	----	3479- 3363	3060	1697	1678	1618	1562- 1307	255	570	469- 435

3.4. The UV-Vis. Spectrum of ligand L1: The appeared bands have dual peaks; one has been in (284 nm) attributable to electronic transition of ($\pi \rightarrow \pi^*$). The other one is observed in 391 nm because of electronic transition of ($n \rightarrow \pi^*$) [16,25]. Table (5) has given details of electronic spectrum data the μ_{eff} values of **La** (5.63BM.). Table (5) has presented five absorption peaks for UV-Vis bands of Mn(II). The absorption peaks at (275 nm) is as a result of intra ligand. In (367 nm), the peak is assigned to intra ligand with charge transfer (C.T.) analogous to (L1) band. At (410 nm), the peaks have been specified to (C.T.). At (545 nm) and (769, 953 nm), the peak is allocated for (d-d) electronic transition of ${}^6A_{1g} \rightarrow {}^4T_{2g}(G)$ and ${}^6A_{1g} \rightarrow {}^4T_{2g}(D)$. Electronic peaks of spectrum have submitted an octahedral geometrical structure for Mn(II) ion. The μ_{eff} values of **Lb** (3.9BM.), and UV-Vis band of Co(II) complex, have depicted six

Absorption peaks. At (275 nm), the absorption peak is related with intra ligand comparably with (L₁) spectrum. At (345 nm), the peak has been owing to intra ligand with charge transfer (C.T.). At (400 nm), the peak is in accordance with charge. At 697 , 822 and 859, newfangled 3 absorbing peaks with weak intensity have been as a consequence of (d-d) electronic transition type $^4T_{1g}(F) \rightarrow ^4A_{2g}(F)$ and $^4T_{1g}(F) \rightarrow ^4T_{2g}(F)$. Those peaks have been typical in the position with described octahedral Co(II) as stated in[25]. The μ_{eff} values of **Lc** , and the UV-Vis spectrum of Ni(II) complex, have presented 5 absorbing peaks. At (280 and 391 nm), dual absorption peaks are related with intra ligand with respect to (L₁) spectrum. However, at 447 nm, the peak has been attributable to C.T. At (769 and 793 nm), newfangled two absorbance peak of weak intensity have been in accordance with (d-d) electronic transition type $^3A_{2g}(F) \rightarrow ^3T_{1g}(F)$. Those peaks are comparable to others octahedral Ni(II)[25]. The μ_{eff} values of **Ld** complex (1.72BM.), the UV-Vis spectrum of Cu(II) complex, displayed four absorbance peaks. At (297 and 342 nm), dual absorption peaks have been in accordance with intra ligand with (L₁) spectrum. At (495 nm), the peak has been as a result of C.T. At (860 nm), newfangled peak has been observed as result of (d-d) electronic transition type $^2E_g \rightarrow ^2T_{2g}$. They have similarity in location with reported studies of octahedral Cu(II) . The UV-Vis spectrum of **Le** and **Lf** complexes, This spectrum has shown absorption peaks, at (281 , 345 and 410 nm) and peaks at (281 , 345 and 415nm) are in accordance with intra ligand with L₁ spectrum along with (C.T) correspondingly. These peaks possess resemblance in the position with octahedral Cd (II) as well as Hg(II) reported in [25,26].

Table 5. Electronic Data of ligand and their Complexes

Comp.	λ_{\max}	$\bar{\nu} \text{ cm}^{-1}$	$\epsilon \cdot \text{mol}^{-1} \cdot \text{L} \cdot \text{cm}^{-1}$	Assignments	μ_{eff} (BM)
L1	284	35211	2464	$\pi \rightarrow \pi^*$	---
	391	25575	624	$n \rightarrow$	
	275	36363	2177	Intra-ligand	
La	367	27247	1513	Intra-	5.63
	410	24390	250	ligand+C.T	
	545	18345	21	C.T.	
	769	13003	19	${}^6A_1g \rightarrow$	
	953	10493	17	${}^4T_2g(G)$	
	275	36363	2196	Intra-ligand	
Lb	345	28985	1965	Intra-ligand +	9.3
	400	25000	250	C.T C.T.	
	697	14347	15	${}^4T_1g(F) \rightarrow$	
	822	12165	10	${}^4A_2g(F) \nu_2$	
	859	11641	10	${}^4T_1g(F) \nu_1$	
	280	35714	2450	Intra-	
Lc	391	25575	548	ligand	4.85
	447	22371	230	Intra -	
	769	13003	19	ligand	
	793	17610	19	C.T.	
	297	33670	2454	Intra-	
Ld	342	29239	2354	ligand	1.72
	495	20202	130	Intra-	
	860	11627	7	ligand	
Le	281	35587	2406	Intra-	Dia.
	345	28985	1757	ligand	
	410	24390	405	Intra-	
Lf	281	35587	2351	Intra-	Dia.
	345	28985	1934	ligand	
	415	24096	350	Intra-	

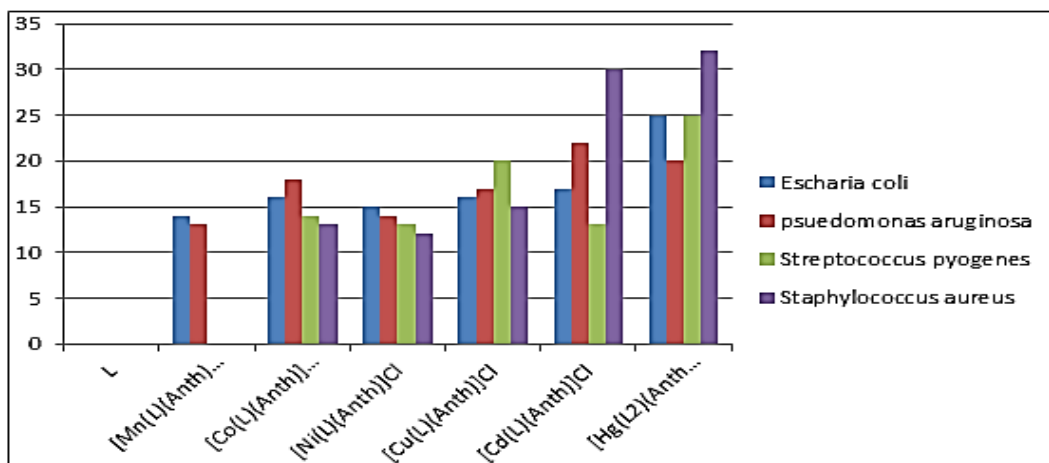
3.5. Antibacterial Activity:

By the use of well plate technique in nutrient agar, formed L ligands and their metal compounds in this paper have been investigated in contradiction of 4 categories of (+) bacteria gram and dual gram (-)[27-29]. The (ZI) of the metallic (L) complexes in contradiction of bacteria growth has been itemized in table (6). Accordingly, the results of organic **La** complex activity toward bacteria number 1 and 2 has been satisfactory under ZI=13,14 mm. But, it wasn't exposed any behavior for bacteria number 3 and 4 under ZI=zero. The (**Lb**) complex has been detected by the identical behavior for *Streptococcus pyogenes* and *Staphylococcus aureus*

(ZI=13mm). However, the organic behavior concerning *E.coli* as well as *psuedomonas aruginosa* was with ZI=18 and 16mm. The **(Lc)** has exposed worthy performance for each bacteria kind over than ligand (L). The uppermost organic behavior for **(Ld)** complex was in the direction of *Streptococcus pyogenes* (ZI=20 mm). But, the maximum organic behavior for **(Le)** complex was *Staphylococcus aureus* (ZI=30 mm). As compared with complexes of (L₁), the results of mercury complex **(Lf)** was given the utmost organic performances (ZI=32,25,20 and 25 mm) for entire bacteria categories.

Table 6. The Inhibitingzone (IZ) for all compounds

	1	2	3	4
L₁	Zero	zero	Zero	zero
La	13	14	Zero	zero
Lb	18	16	13	13
Lc	14	15	12	13
Ld	17	16	15	20
Le	22	17	30	13
Lf	20	25	32	25



Chat (1) : (ZI) mm of ligand and their Complexes

4. Conclusions

In the present study, new Schiff base derived from 5-Amino-2-phenyl-2,4-dihydro-pyrazol-3-one with o-phthalaldehyde was synthesized and characterized. The results showed that Schiff base ligand format with metal ions through the N2 worked were as donor atoms. The results of the electron spectra and the magnetic susceptibility of tetrahedral geometry of all complexes are presented. The composite compounds were studied as antimicrobial and the results showed that all complexes have a various activities against bacteria .

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