

Preparation, Characterization and Biological Study of New Boron Compound and Schiff Base Derived From 2-Aminophenol with Their Cu (II) and Pt (IV) Complexes

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Abstract: Two new ligands derived from 2-aminophenol have been prepared. The first ligand (boron compound) L_1 was prepared by refluxing one mole of sodium borohydride with two mole of 2-aminophenol in a mixture of methanol: acetone (2:1). The second ligand (Schiff base) L_2 was prepared by refluxing equimolar from 4-bromoacetophenone and 2-aminophenol in methanol: acetone (2:1). Cu (II) and Pt (IV) complexes of ligands have been also prepared. The ligands and their metal complexes were characterized by elemental analysis, molar conductivity and spectral studies of FTIR, ^1H , ^{13}C NMR, thermal analysis, UV-VIS, as well as atomic absorption and magnetic susceptibility of metal complexes. All prepared compounds were tested as antibacterial agents against gram negative bacteria (*E-coli*) and gram positive bacteria (*Staphylococcus aureus*).

Keyword: 2-aminophenol, boron compounds, Schiff bases.

I. Introduction

A large number of Schiff bases and their complexes have been studied for their interesting and important properties [1]. The great interest in Schiff bases derived from 2-aminophenol is due to biological importance for having antidepressant, antimicrobial, antitumor, antiphlogistic, nematocidal, and other medicinal agents [2]. Many Schiff base ligands of 2-aminophenol have been synthesized by condensation of 2-aminophenol with phthalaldehyde[3], thiazinediketone[4], salicylaldehyde[5], 2,6-pyridinedicarboxaldehyde[6], terephthalaldehyde[7], pyrrol-2-carboxaldehyde[8], 4-aminoantipyrine[9], 1,4-diformyl benzen[10], 3-phenylazo and thiazolylazopentadion[11], dicinnamoylmethane[12], 4-fluorophenyl-1-phenyl-pyrazol-4-carboxaldehyde[13]. Boron is a natural product and occurs in the environment as borates [14]. Many natural products comprise boron isolated from bacteria, such as the antibiotic boromycin [15]. These natural products show that boron can be used in biological field without negative results for human health [16]. Boron compounds have unique physical and chemical properties such as low toxicity and thermal stability, therefore these compounds were used in a wide range of applications such as reagent materials, catalysts also used in medicine, radioactive metals, polymer science and supramolecular chemistry. The complexes of boron can be used for destroying diseased cell [17,18]. This paper reports the preparation and biological study of new two compounds derived from 2-aminophenol, the boron compound of 2-aminophenol and Schiff base of 2-aminophenol with 4-bromoacetophenone as well as their copper (II) and platinum (IV) complexes. The biological activity for all prepared compounds were compared with starting materials.

II. Experimental

2.1- Materials, Instruments and Methods

All chemicals used were of analytical reagent grade. FTIR spectra were recorded on SHIMADZU 8400s spectrophotometer. Thermal analysis (TG and DTG) were carried out by using METTLER TA4000 SYSTEM. The electronic spectra (190-1100 nm) in DMSO were recorded on (SHIMADZU 1800-UV spectrophotometer). Elemental microanalysis were performed on (CHNS Elemental Analyzer CHNS-932). ^1H NMR and ^{13}C NMR were recorded on (Bruker NMR spectrometer 400 MHz Avance III 400). The metal contents of the complexes were measured by atomic absorption technique using (Nov AA 350 spectrophotometer). Electrical conductivity measurements for complexes (10^{-3}M) in DMSO at room temperature were carried out by using Hunts Capacitor Trade Mark British made. The chloride content for complexes was determined by Mohr's method. Magnetic moments (μ_{eff} , B.M) for the prepared complexes in the solid state at room temperature were measured by (Auto Magnetic Susceptibility Balance Model Sherwood Scientific).

2.2-Synthesis of 2-boraneamino phenol (L_1).

2-aminophenol (0.2g, 1.834mmol) in a mixture of methanol:acetone (2:1) was mixed with a solution of sodiumborohydride (0.034g, 0.917mmol) in 4.5 ml of methanol. The reaction mixture was heated under reflux for 12 h under dry condition, evolved hydrogen gas was collected over water. The precipitation of the product

took place after evaporating part of the solvent followed by addition of ether giving brown powder. The product was washed with ether several times and dried in air.

2.3-Synthesis of 2-((1-(4-bromophenyl)ethylidene)amino)phenol (L₂).

A hot solution of 4-bromoacetophenone (0.456g, 1.83mmol) in 6 ml methanol was added to a hot solution of 2-aminophenol (0.2g, 1.83mmol) in a mixture of methanol:acetone (2:1) with 1 ml of glacial acetic acid as catalyst and the reaction mixture was heated under reflux for 16 h. Precipitation of the product took place after evaporating part of the solvent followed by addition of ether giving golden brown precipitate. The product was washed several times with ether and dried in air.

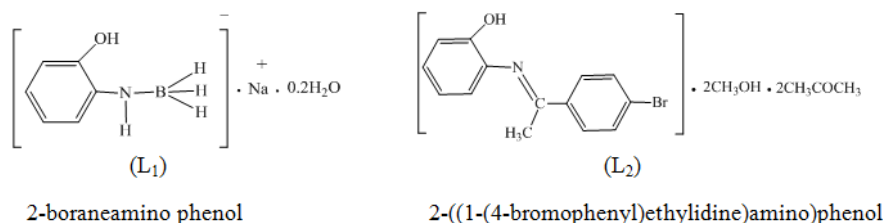


Figure (1) : The structures of synthesized ligands L₁ and L₂.

2.4-Synthesis of Copper Complexes C₁ and C₃

Cu(II) complex C₁ was prepared by adding a solution of CuCl₂·2H₂O (0.0574g, 0.3368mmol) in 4 ml methanol to a hot solution of L₁ (0.1g, 0.6737mmol) in a mixture of methanol:acetone (2:1). The reaction mixture was heated under reflux for 4 h. Part of solvent was evaporated and the dark brown product was obtained after addition of ether, washed several times with ether and dried in air. Cu(II) complex C₃ was prepared by the same method mentioned in preparation of C₁, using L₂ (0.1g, 0.2128mmol) and CuCl₂·2H₂O (0.0181g, 0.1064mmol).

2.5-Synthesis of Platinum Complexes C₂ and C₄

Platinum complexes were prepared by the same method mentioned in preparation of C₁, using 0.1g of L₁ or L₂ (0.6737 and 0.2128mmol) respectively with K₂PtCl₆ (0.1637g, 0.3368mmol) in 22 ml DMSO for C₂ and (0.0517g, 0.1064mmol) in 6 ml DMSO for C₄ respectively.

III. Results And Discussion

The physical and analytical data (Table 1) are agreement with suggested structures of studied compounds.

Table (1) : The physical properties and analytical data for two ligands and their metal complexes

Symbol	Color	Yield%	m.p ^o C	CHN analysis Found(calc.)			M% Found(calc.)	Cl% Found(calc.)
				C%	H%	N%		
L ₁	Brown	70.2	>250	49.15 (48.51)	6.1 (6.3)	8.97 (9.43)	–	–
L ₂	Golden-brown	21.9	134-137	56.27 (56.18)	5.95 (6.80)	4.34 (2.97)	–	–
C ₁	Dark brown	79.3	>360	35.75 (36.60)	5.13 (6.10)	5.21 (4.27)	–	9.62 (10.86)
C ₂	Dark brown	33	>360	37.85 (37.28)	4.81 (4.94)	6.56 (5.93)	27.48 (27.55)	3.2 (5.01)
C ₃	Dark brown	48.3	>360	52.53 (51.92)	3.57 (3.70)	3.82 (4.32)	10.88 (9.87)	2.6 (2.7)
C ₄	Dark brown	39.2	>360	24.42 (23.43)	2.55 (3.06)	1.59 (1.95)	–	2.8 (4.5)

3.1-IR spectra

The FTIR spectra of ligands and their metal complexes were studied and the data are given in Table (2). The lattice and coordinated water were observed at (3420-3404) and 759 cm⁻¹ [19,20]. The spectra of the first ligand (L₁) (Figure 2) showed a strong band at 3400 cm⁻¹ due to stretching vibration of O-H group, this band was shifted to the lower frequency in the spectrum of C₁ complex at 3379 cm⁻¹ as a result of complexation. [21] The band appeared at 3377 cm⁻¹ was attributed to stretching vibration of N-H group for L₁ and which is absent in spectra of C₁ and C₂ complexes because of coordination through the N atom. [22] The spectra of L₁, C₁ and C₂ showed bands at 2366, 2366 and 2363 cm⁻¹ respectively, these bands assigned to vibrational mode of B-H. [23] The spectrum of L₁ exhibited the moderate band at 1454 cm⁻¹ belong to ν_{B-N}, while this band was shifted to lower frequency at 1430 and 1417 cm⁻¹ for C₁ and C₂ respectively. [23] The strong band at 3448 cm⁻¹ is due to ν_{O-H} (phenolic and methanolic) for L₂ (Figure 2), this band shifted to 3400 cm⁻¹ in C₄ complex because of complexation [21,24]. The band appeared at 1581 cm⁻¹ refer to stretching mode of C=N in L₂, this band change in

profile and shifted to lower frequency at 1583 and 1577 cm^{-1} in C_3 and C_4 respectively as a result of coordinate N atom of $\text{C}=\text{N}$ with metal ions.^[19,21,25] Bands appeared at lower frequency due to stretching vibration of $\text{M}-\text{O}$, $\text{M}-\text{N}$ and $\text{M}-\text{Cl}$.^[21]

Table (2):IR Spectra of two ligands and their metal complexes

compound	$\nu\text{O}-\text{H}$	$\nu\text{N}-\text{H}$	$\nu\text{B}-\text{H}$	$\nu\text{B}-\text{N}$	$\nu\text{C}=\text{N}$	H_2O lattice (coordinate)	$\nu\text{M}-\text{O}$	$\nu\text{M}-\text{N}$	$\nu\text{M}-\text{Cl}$
L1	3400	3377	2366	1454	-	3417	-	-	-
C1	3379	-	2366	1430	-	(3404,759)	600	370	343
C2	3404	-	2363	1417	-	-	530	341	273
L2	3448	-	-	-	1581	-	-	-	-
C3	3440	-	-	-	1583	-	-	387	312
C4	3400	-	-	-	1577	3420	500	435	318brg.

Brg.= bridging bond.

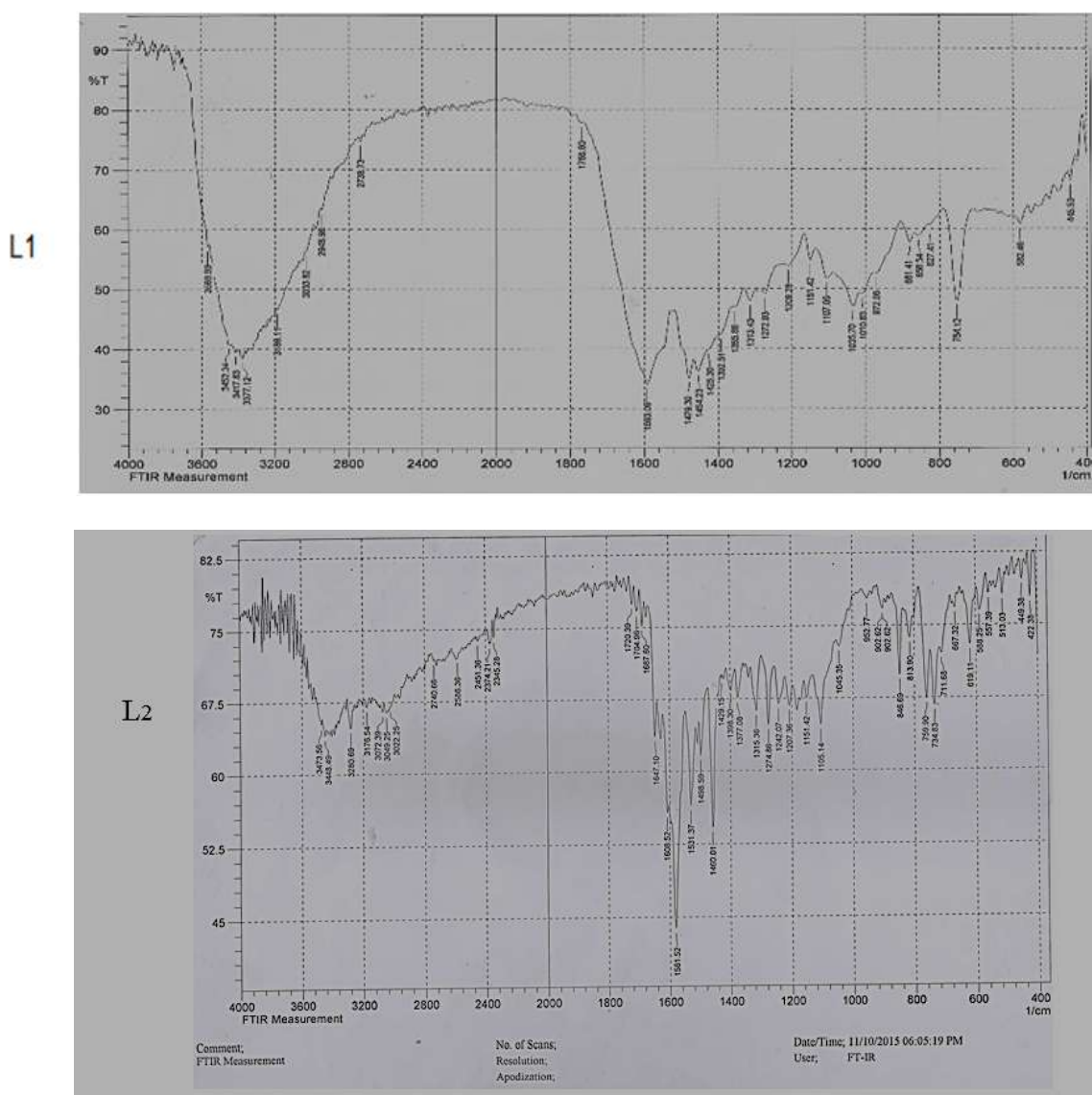


Figure (2):FTIR Spectra of L_1 and L_2 .

3.2-Thermal Analysis

The thermal stability of ligands were studied by TG and DTG analysis. The thermogram of L₁ exhibited two steps at temperature range (29.3-181°C) and (181-774.7°C) with residue. Four steps appeared in thermogram of L₂ at temperature range (33.7-172.3°C),(172.3-280.7°C),(280.7-354.3°C) and (354.3-601.3°C). The first step for each ligands involved loss of solvents and H₂O and this due to their low boiling points^[26,27]. The data (Table 3) showed the agreement between the theoretical and practical fragments and the results exhibited more stability of L₁ than L₂.

Table (3) : Thermal decomposition of L₁ and L₂.

Compound	Steps	Temp. range of Decomposition °C	% weight loss Found (calc.)
L ₁ C ₆ H ₉ NOB.Na.0.2H ₂ O 4H M.wt= 148.42	0.2 H ₂ O	29.3-181.0	17.77 (16.57)
	OH		
	4H	181-774.7	60.16 (60.63)
	C ₆ H ₄ N	-	21.66(22.70)
L ₂ C ₆ H ₁₂ NOBr.2CH ₃ OH .2CH ₃ COCH ₃ 8 H M.wt= 469.9	2 CH ₃ OH	33.7-172.3	38.83 (40.00)
	2 CH ₃ COCH ₃		
	8 H	172.3-280.7	46.48 (47.64)
	C ₆ Br + C ₆	280.7-354.3	3.17 (2.55)
	C	354.3-601.3	11.53 (9.78)
	CH ₃ + N + OH		

3.3-¹HNMR Analysis

The peak assignments of ¹HNMR are described in Table (4). The ¹HNMR spectra of L₁ and L₂ in DMSO (Figure 3) exhibited broad and moderate multiplet signals of aromatic protons at δ (6.35-7.7 ppm).^[28] The OH proton showed chemical shift at δ 8.5 and 8.9 ppm for L₁ and L₂ respectively.^[24] The spectrum of L₁ showed broad peak appeared at δ 3.4 ppm which was assigned to protons of B-H and H₂O as well as dissolved water in DMSO^[23]. The chemical shift of amine proton was observed at δ 8.3 ppm^[29]. The spectrum of L₂ exhibited peak at δ 1.9 ppm which was assigned to protons of methyl group (CH₃ group and CH₃ in acetone), While the proton of methyl group in methanol shifted to δ 3.3 ppm.^[30,31,32] Also the dissolved water in DMSO was appeared at δ 3.3 ppm^[23]. A weak peak was observed at δ 4.3 ppm which was assigned to proton of methanolicOH^[30]. The strong peak at δ 2.5 ppm was attributed to residual DMSO^[23,30].

Table (4): Chemical shifts for ¹HNMR of L₁ and L₂.

L ₁			L ₂		
Chemical shifts δ(ppm)	Mark	Assignments in DMSO	Chemical shifts δ(ppm)	Mark	Assignments in DMSO
3.4,3H,s	9,10	B-H ,H ₂ O protons and dissolved water in DMSO	1.9,9H,s	8,16,18	CH ₃ protons and CH ₃ in acetone protons
6.35-7.7,4H,m	3-6	Aromatic protons	3.3,3H,s	15	Methanolic CH ₃ protons and dissolved water in DMSO
8.3,1H,s	7	N-H proton	4.3,1H,s	15	Methanolic OH proton
8.5,1H,s	2	O-H proton	6.35-6.63,8H,m	3-6 10,11,13,14	Aromatic protons
			8.9,1H,s	2	O-H proton

S=single , m= multiple

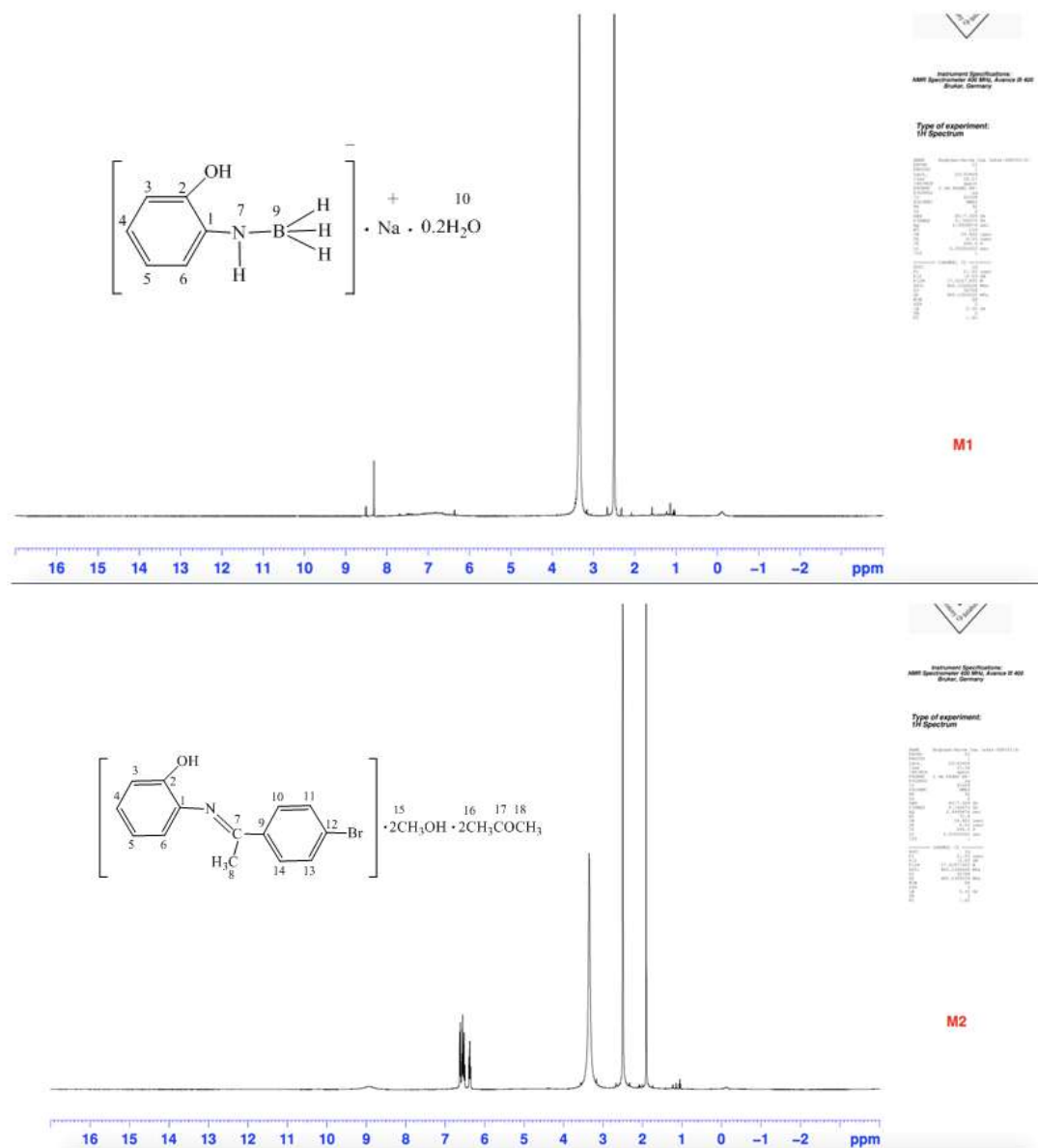


Figure (3) : ^1H NMR spectra of L_1 and L_2 .

3.4- ^{13}C NMR Analysis

The peak assignments of ^{13}C NMR are shown in Table (5). The ^{13}C NMR spectra of ligand L_1 and L_2 in DMSO showed resonance bands at δ (114.5-137.0 ppm) were assigned to aromatic carbon.^[19] The carbon atoms number 1 gave peaks at δ 125 and 130.4 ppm for L_1 and L_2 respectively, while the carbon atoms number 2 have been shifted to δ 145.5 and 144 ppm for L_1 and L_2 respectively.^[30,33] The L_2 spectra showed chemical shift of methyl group at δ 20 ppm.^[19] The peak observed at δ 172 ppm was assigned to azomethine carbon^[19]. The carbon atom of methanol (C-O) was exhibited peak at δ 49 ppm, and the carbon atom in acetone (C=O) showed a peak at δ 204 ppm^[30], while the carbon atoms number 16 and 18 were exhibited peak at 34 ppm.^[30] The chemical shift of DMSO was appeared at δ 40 ppm.^[30]

Table (5): Chemical shifts (ppm) for ^{13}C NMR of L_1 and L_2 .

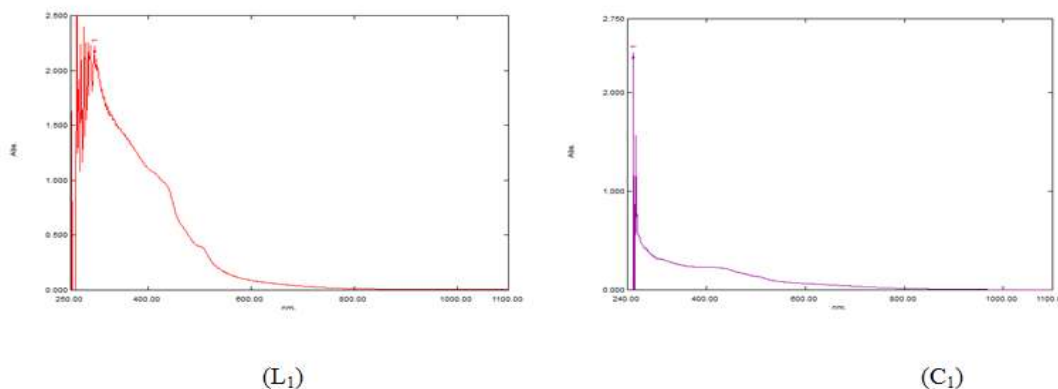
L_1			L_2		
Chemical shifts δ (ppm)	Mark	Assignment in DMSO	Chemical shifts δ (ppm)	Mark	Assignment in DMSO
127-137	3-6	Aromatic carbons	20	8	Carbon of methyl group
125	1	Carbon of amine	34	16,18	Carbon of acetone
145.5	2	Carbon of phenol	49	15	Methanolic carbon
			114-125.2	3-6 9-14	Aromatic carbons
			130.4	1	Carbon of amine
			136.3	12	Carbon of brom
			144	2	Phenolic carbon
			172	7	Carbon of azomethine
			204	17	Carbon of acetone

3.5-Electronic spectra

The spectral data of two ligands and their metal complexes in DMSO are given in Table (6). The spectra of L_1 (Figure 4) and L_2 exhibited high intensity bands at 33783 and 40650 cm^{-1} of L_1 and L_2 respectively, which were assigned to $\pi \rightarrow \pi^*$ transitions, in addition, low intensity bands appeared at 24390 and 23980 cm^{-1} of L_1 and L_2 respectively which were assigned to $n \rightarrow \pi^*$ transitions. The spectra of L_1 complexes exhibited bathochromic shift of ligand band to 39682 and 45454 cm^{-1} for C_1 and C_2 respectively, while the complexes of L_2 showed hypsochromic shift of ligand band to 34482 and 39062 cm^{-1} for C_3 and C_4 respectively. The spectrum of Cu (II) complex C_1 (Figure 4) showed absorption bands in the region around 15825 and 19801 cm^{-1} and were attributed to ${}^2\text{B}_{1g} \rightarrow {}^2\text{B}_{2g}$ (ν_1) and ${}^2\text{B}_{1g} \rightarrow {}^2\text{E}_g$ (ν_2) respectively of squar planer copper (II) complexes^[19]. The magnetic moment of copper complex C_1 was $\mu_{\text{eff.}} = 1.74$ B.M and this value agree with squar planer geometry^[19,22]. The band observed at 21190 cm^{-1} belong to charge transfer transition. The spectrum of copper complex C_3 exhibited three bands at 15329, 19794 and 20976 cm^{-1} and these bands were assigned to ${}^2\text{B}_{1g} \rightarrow {}^2\text{A}_{1g}$ (ν_1), ${}^2\text{B}_{1g} \rightarrow {}^2\text{B}_{2g}$ (ν_2) and ${}^2\text{B}_{1g} \rightarrow {}^2\text{E}_g$ (ν_3) transitions respectively of tetragonally distorted octahedral copper (II) complexes^[19]. The magnetic moment of copper complex C_3 was $\mu_{\text{eff.}} = 2.4$ B.M and this value attributed to octahedral geometry^[20]. The spectrum of diamagnetic platinum complex C_2 exhibited bands at 19801 and 24103 cm^{-1} which was belong to ${}^1\text{A}_{1g} \rightarrow {}^3\text{T}_{1g}(\text{H})$ and ${}^1\text{A}_{1g} \rightarrow {}^3\text{T}_{2g}$ transition of octahedral geometry^[34]. The spectrum of diamagnetic platinum complex C_4 exhibited three bands at 12195, 22935 and 29614 cm^{-1} due to ${}^1\text{A}_{1g} \rightarrow {}^3\text{T}_{1g}(\text{H})$ (ν_1), ${}^1\text{A}_{1g} \rightarrow {}^3\text{T}_{2g}$ (ν_2) and ${}^1\text{A}_{1g} \rightarrow {}^3\text{T}_{1g}$ (ν_3) transitions respectively of octahedral Pt(IV) complex^[34]. The conductivity measurements showed that all studied complexes were non electrolyte.^[35]

Table (6): Electronic spectra, magnetic moment and molar conductance with suggested structures of L_1 and L_2 complexes.

symbol	Band position cm^{-1}	Assignments	$\mu_{\text{eff.}}$ B.M	Molar Conductivity $\text{S.mol}^{-1}.\text{cm}^{-1}$	Suggested structures
C_1 Cu(II)	ν_1 15825 ν_2 19801 ν_3 21190	${}^2\text{B}_{1g} \rightarrow {}^2\text{B}_{2g}$ ${}^2\text{B}_{1g} \rightarrow {}^2\text{E}_g$ $\text{L} \rightarrow \text{M}(\text{C.T})$	1.74	0.0681	Squar planer
C_2 Pt(IV)	ν_1 19801 ν_2 24103	${}^1\text{A}_{1g} \rightarrow {}^3\text{T}_{1g}(\text{H})$ ${}^1\text{A}_{1g} \rightarrow {}^3\text{T}_{2g}$	diamagnetic	0.1077	octahedral
C_3 Cu(II)	ν_1 15329 ν_2 19794 ν_3 20976	${}^2\text{B}_{1g} \rightarrow {}^2\text{A}_{1g}$ ${}^2\text{B}_{1g} \rightarrow {}^2\text{B}_{2g}$ $\text{L} \rightarrow \text{M}(\text{C.T})$	2.4	0.0337	distorted octahedral
C_4 Pt(IV)	ν_1 12195 ν_2 22935 ν_3 29614	${}^1\text{A}_{1g} \rightarrow {}^3\text{T}_{1g}(\text{H})$ ${}^1\text{A}_{1g} \rightarrow {}^3\text{T}_{2g}$ ${}^1\text{A}_{1g} \rightarrow {}^3\text{T}_{1g}$	diamagnetic	0.0591	octahedral

**Figure (4):** UV-VIS spectra of L_1 and its copper complex C_1 .

3.6- Antibacterial Activity

Screening for antibacterial activity of all studied compounds and starting materials were tested against G –ve bacteria (*E-Coli*) and G +ve bacteria (*Staphylococcus aureus*) by agar diffusion method using 1×10^2 M of studied compounds in DMSO. The obtained data Table(7) showed that all compounds were in active against *E-Coli*, while these compounds exhibited different activities against *Staphylococcus aureus* except 4-bromoacetophenon and sodium borohydride were in active. The antibacterial activity of studied compounds were compared with antibiotics cephalaxim and cefatoxim.

Table (7): Antibacterial activity of two ligands and their metal complexes.

Compounds	<i>Staphylococcus aureus</i> diameter mm	<i>E-Coli</i> diameter mm
DMSO	zero	zero
2-aminophenol	36	zero
4-bromoacetophenone	zero	zero
Sodium borohydride	zero	zero
L ₁	28	zero
L ₂	25	zero
C ₁	23	zero
C ₂	20	zero
C ₃	32	zero
C ₄	20	zero
Cephalexim	25	zero
Cefatoxim	48	45

C₁

C₂

C₃ C₄

Figure (5) : suggested structures of the prepared complexes.

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