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Synthesis, Spectral and Bacterial Studies of Mixed Ligand Complexes of Schiff Base Derived from Methyldopa and Anthranilic Acid with Some Metal Ions

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Abstract

Bidentate Schiff base ligand 3-(3,4-Dihydroxy-phenyl)-2-[(4-dimethylamino-benzylidene)-amino]-2-methyl-propionic acid was prepared and characterized by spectroscopic techniques studies and elemental analysis. The Cd(II), Ni(II), Cu(II), Co(II), Cr(III) and Fe(III) of mixed-ligand complexes were structural explicate through moler conductance, [FT-IR, UV-Vis & AAS], chloride contents, , and magnetic susceptibility measurements. Octahedral geometries have been suggested for all complexes. The Schiff base and its complexes were tested against various bacterial species, two of {gram(G+) and gram(G-)} were shown weak to good activity against all bacteria.

Keywords: Bidentate Schiff Base, Methyldopa, Spectroscopic techniques, and bacterial activities.

1. Introduction

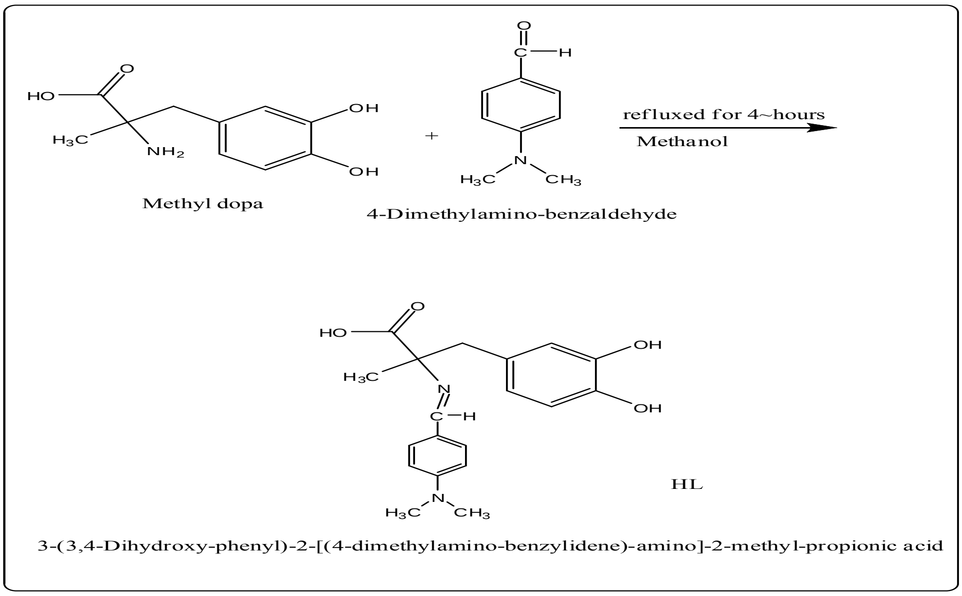
Coordination chemistry that related with the chelate or complexes got a large attention [1-2]. Through chemical bonding as the recent studies the formula which involve of central metal (Lewis acid) and ligands (Lewis base) gives us a strong bond and very stable compounds [3]. Schiff bases form stable complexes with some transition metal ions, and they play an important role in general life with industries such as chemistry and biological activities [4 -6]. Methyldopa, (M-dop) = (α-methyl-3,4-dihydroxyphenyl alanine) is one of the catecholic molecules which are liable to interact with Fe (II). It is a catecholamine used (anti-hypertensive drug) [7]. The stability constants (Ks) of (4 metals) with (Nitrilotriacetic &/ or Iminodiacetic) acids as primary ligands and (Methyldopa and/ or levodopa) as secondary ligands potentiometrically [8]. The spectrophotometric determination of dopamine. HCl and Methyldopa in pharmaceutical preparations using flow injection analysis (FIA). The method is based on oxidative coupling reaction of drug with 2-Furoic acid hydrazide ($C_5H_6N_2O_2$) in the presence of Sodium nitroprusside in (NaOH) medium to form soluble product. The results obtained were in good agreement with those obtained by British Pharmacopoeia method [9]. The stability constants of ternary complexes of M(II) ions with two amini acids (aspartic acid(ASP) &Glutamic (Glu) acid) as primary ligands and (levodopa & Methyldopa)as secondary ligands, also all complexes have been carried out (pH-metrically) [10].

2. Experimental

All chemicals used in this research were purchased from BDH, Fluka and Merck companies and used without moreover purification**.** The melting points were obtained using "Stuart Melting Point Apparatus". The metal contents of the complexes were obtained by atomic absorption technique using a "Shimadzu AA 620G ". The Chloride contents of complexes were obtained by testing all complexes (0.05 gm.) which were decomposed with conc. Nitric acid and diluted with water. The ^{1}H and ^{13}C NMR spectra were performed on by "Brucker DRX system 500 (500 MHz)", University of Tehran. Elemental micro analysis CHNS was carried out by the Euro EA 3000, University of Baghdad. UV-Vis spectra were performed on a "Shimadzu UV- 160A". The FTIR- spectra were carried out by a "Shimadzu, FTIR- 8400S" (4000- 400) cm^{-1} with samples prepared as KBr discs. Magnetic measurements were recorded on a "Bruker BM6 instrument" at 298K following the Faraday's method'.

Ligand's Preparation (HL [11])

A solution of {4-DMBA} 1 mmole in methanol (10 ml) was added to a solution of {M-Dopa} 1 mmole in methanol (20 ml) and then 1mmol of KOH. The mixture was refluxed for 4~hours with stirring. The product was a deep orange solution. It was allowed to cool and dry at room temperature, then recrystallized with ethanol. The brown colored solid mass formed during refluxing was cooled at room temperature, filtered and washed completely with hot ethanol, and recrystallized from acetone to get a pure sample. It yields 90% . The synthetic pathway is as shown in Scheme -1.



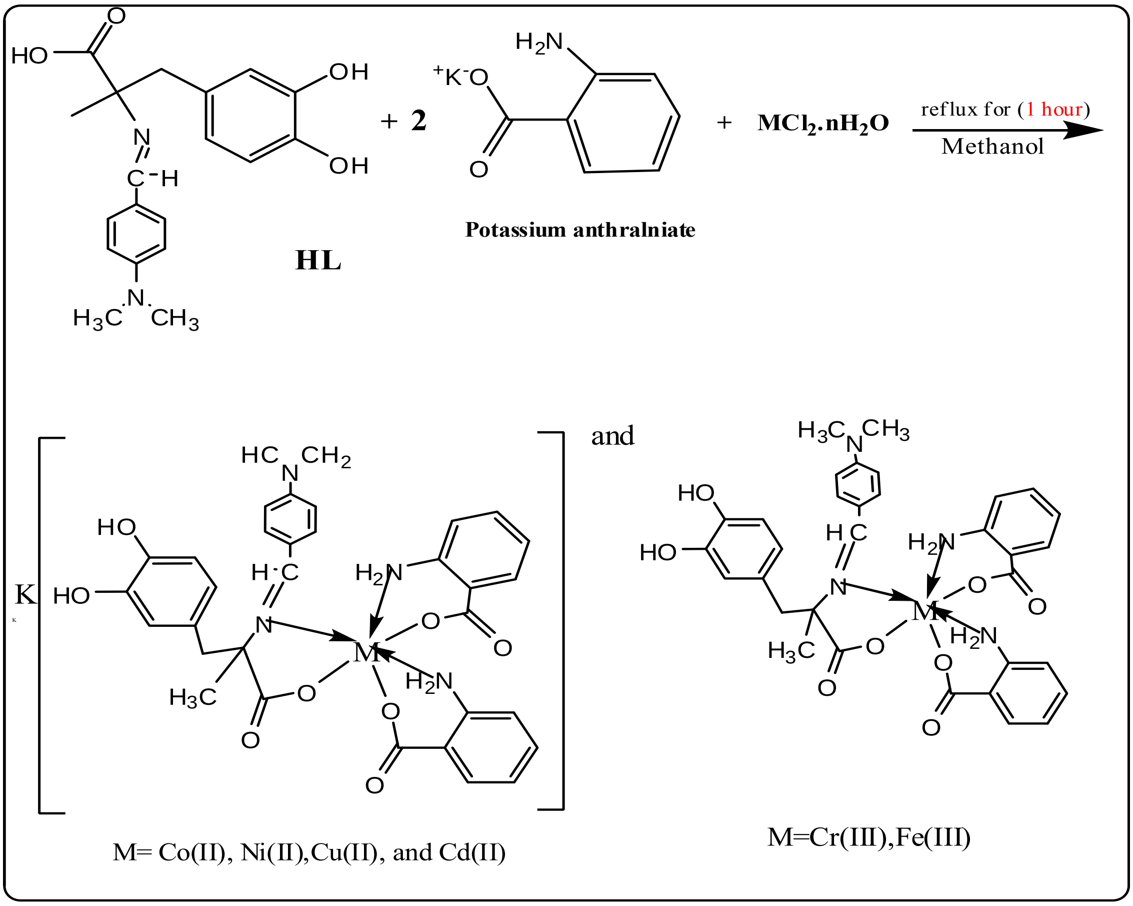
Scheme (1): The preparation of the ligand (HL)

Preparation of potassium anthranilate {$C_7H_6NO_2K$}:

potassium anthranilate ligand has been prepared depending on literature method [11].

A general method in preparation of complexes [11]:

In methanol solution (2mmol of $C_7H_6NO_2K$) and a solution of (HL & KOH) each one is 1mmole were added to a stirred for (1 hour) solution of $MCl_x.nH_2O$ 1mmole. The mixture was (filtered & precipitation) then washed several times with a surplus of ethanol and dried during (24 hours) at room temperature The synthetic pathway is as shown in Scheme -2.



Scheme (2): The preparation of complexes

3. Results and Discussion

1. Physical Properties

- The physical properties for the starting materials and Schiff base (HL) ligand are given in Table (1a and 1b).
- The complexes are soluble in (DMSO) & (DMF), while insoluble in water [12].
- The melting points of ligands were lower than all complexes, Table -1c.
- The conductivity measurement values in DMSO (10^{-3}Molar) solution range in (31.4-38.1) $\Omega^{-1}cm^{2}mol^{-1}$ for Cd(II) ,Ni(II) ,Cu(II) , and Co(II), complexes. the complexes are electrolytes types 1:1 as the data indicate, but the complexes Cr(III)& Fe(III) were non-electrolytes [13].
- Silver nitrate solution was (-) when we exanimated (Chloride ion)
- The found and theory values of (M%) in each complex are in good agreement [14].

2. ^{1}H-NMR spectrum for the ligand (HL)

The integral intensities (δ) in ppm of each signal in the ^{1}H-NMR spectrum of HL Figure-1, was found to agree with the number of different types of protons present. The signal obtained in range δ (6.44 -7.68) ppm was appointed for doublet due one proton of aromatic ring of phenyl, the formation of Schiff base is supported by the presence of a singlet at (δ 8.46) ppm corresponding to the azomethine proton

(–N=CH). The signal obtained in range δ (7.10-7.70) ppm was appointed for doublet due one proton of aromatic ring. (m, arom. proton,). The signals observed at δ (1.31 & 1.23) ppm ascribed to methyl protons (-CH_3) group4[15,16].

3. ^{13}C-NMR Spectrum for (HL)

The spectrum of [HL] in (DMSO-d_6 solvent) is shown in Figure-2. Chemical shift [δ ppm] azomethine H-C=N carbon (C1) at 195ppm. The carbon atoms of aromatic ring at range (δ=130.25-139.24) ppm, note methyl carbon (3CH_3) in Schiff base at (δ=41.72 ,41.51 and 41.09) and at δ=40.68 ppm is ascribed to methyl carbon (1CH_3) of DMSO [15,16].

4. FTIR spectra:

Methyl dopa spectrum: The bands at (1490) cm^{-1}, (3105) cm^{-1}, (2956, 2808) cm^{-1} and (1257) cm^{-1} were appointed to υ(C=C) aromatic, υ(C–H) aromatic, υ(C–H) aliphatic stretching vibration and υ(C–C) aliphatic respectively, Table-2a. The band at (1209) cm^{-1} which account for υ(C-N) cm^{-1} stretching vibration. The observed bands at [1618 and 1402]cm^{-1}were due to $υ_{asym}${COO−}carboxyl] and $υ_{sym}${COO−}carboxyl groups,Δυ{COO-}$_{asym}$-υ{COO-}$_{sym}$ =216 cm^{-1}.The strong broad bands around (3481 , and 3421) cm^{-1} were ascribed to the stretching vibration of υ (O-H) .The band at (3223) cm^{-1} was appointed to the stretching vibration of υ (N-H_2) [17,18].

Ligand (HL) spectrum: A very strong broad band around (3473) cm^{-1} ascribed to stretching vibration of (O-H) group, Table-2a and Figure-3. The spectrum shows a new band at (1620) cm^{-1} ascribed to υ(C=N) str. vibrations of the (HL) with disappearance of the stretching vibration bands for the amine (NH_2) group [17]. The bands at (1446) cm^{-1}, (3039) cm^{-1}, (2966) cm^{-1} and (1273)cm^{-1} were appointed to υ(C=C) aromatic, υ(C–H) aromatic, υ(C–H) aliphatic and υ(C–C) aliphatic str. vibration, respectively. The observed bands at carboxyl groups] in (HL) respectively [19,20]. A sharp band at (1620) cm^{-1} was due to stretching vibration of the υ(HC=N-), it was shifted to lower frequency for every complex and its range appeared in (1604–1616) cm^{-1}, indicating coordination of the azomethine N atom (HC=N:→ M) with the metal ion [21] .

Anthranilic acid spectrum : the region of all amino acids (υ NH^{3+}) appears at (3030-3130) cm^{-1} [22], and, the band of AnthH appears at (3101) cm^{-1}, but this band was disappeared in every complex with appearance of coordinated bands NH_2 within the range (3062-3371)cm^{-1}, Table-2b. the bands at (1662 & 1485) cm^{-1} respectively due to υ (COO^-)$_{asym}$ and υ (COO^-)$_{sym}$, therefore Δυ = 177 cm^{-1} , in complexes were shifted to a lower frequency in range (1554-1593)cm^{-1} for υ (COO^-)$_{asym}$ and (1327-1400) cm^{-1} for υ (COO^-)$_{sym}$, Table-2c . The Δυ = [υ (COO-)$_{asym}$ - υ (COO-)$_{sym}$] appeared within the range (189-227) cm^{-1} indicating that the carboxlate ion coordinates as a mono dentate donor with the metal ions [23]. IR spectra explain that the anthranilato ligand ($Anth^-$) is negatively charged can be coordinated to the metal atom [M(II) & M(III)] through the nitrogen atom in amin group (H_2N→M), and oxygen carboxylate group (-COO ⁻M), serve as a bidentate .FT-IR spectral results provide strong evidences for the complexation of Schiff base (HL) with metal ions in bi dentate mode fashion (NO) occured through the [(O) of carboxylato (-COO-) and (N) of the (HC=N-)] groups. New weak intensity bands were observed in the regions (532-586) cm^{-1} might be due to M-N and (412-486) cm^{-1} due to M-O vibrations, [24,25]. The experimental data suggest that the anthranilic acid acts as bidentate uni negative charge and adopt an octahedral geometry as proposed.

5. Electronic spectra and magnetic moment for the ligands and complexes:

The UV-Vis spectrum of the ligand (HL), Figure-4, Table-3 shows two peaks at λ_{max} (301 and 344)nm due to ($\pi\rightarrow\pi^*$) & ($n\rightarrow\pi^*$) transition respectively[26]. The UV-spectrum of (AnthH) showed two high intensity peaks at (242 and 332) nm appointed to ($\pi\rightarrow\pi^*$) & ($n\rightarrow\pi^*$) respectively [22,24].

Cr(III) complex: The (UV- Vis) spectrum, shows high broad peak at 342 nm was indicated to the charge transfer and overlap with υ_3 ($^4A_{2g}\rightarrow{}^4T_{1g}(P)$, while the weak broad peak at 798 nm , which indicated to ($^4A_{2g}\rightarrow{}^4T_{2g}(F)$ (d–d), transition in an octahedral geometry , and Magnetic moment value is 3.94 B. M. indicating three unpaired electrons close to spin only value, that agreement of the structure of Cr (III) to be an octahedral geometry Table-3, [26].

[Fe (L)(Anth)$_2$] complex: The electronic spectrum**,** shows absorption peak in the UV region at 344nm which is ascribed to (CT) transitions while another absorption peak in the visible region at 790 nm which belongs to electronic transition $^6A_1g\rightarrow{}^4T_1g(4P)$, Table-3 [26], and μ_{eff} = 5.09 B.M. is lower than the μ_{eff} of the high spin octahedral complex , which is in agreement with an octahedral geometry [27].

[Co(L)(Anth)$_2$] complex: The (UV- Vis) spectrum, shows more than one peak. The first high intense peak at 344nm which is ascribed to (C.T) transitions. The second peak is at 430 nm which is ascribed to $^4T_1g\rightarrow{}^4T_1g(P)$ υ_3 , and the third absorption peaks are of (d-d) transitions as shown in Table-3, at (734 nm) and 790 nm, which are considered as $^4T_1g\rightarrow T_2g(F)$ υ_1 transitions[28] , and μ_{eff} = 4.36 which suggest an octahedral geometry [24].

[Ni (L)(Anth)$_2$] complex: The electronic spectrum shows two peaks at (344nm), which may be appointed to (CT) transitions & (υ_3) $^3A_2g(F)\rightarrow{}^3T_1g(P)$. The peak at 797 nm, which is due to $^3A_2g(F)\rightarrow{}^3T_1g(F)$ (υ_2) {d-d transitions}, respectively [28], and The μ eff = 2.83 B.M. which suggest an octahedral geometry [24].

[Cu (L)(Anth)$_2$] complex: This complex , shows a high intense peak at 344 nm is ascribed to a strong charge transfer and peak at 696nm due to $^2Eg\rightarrow{}^2T_2g$ .These peaks are characteristic in position and width with those reported for octahedral Copper (II) complexes, [28], and μeff = 1.62 B.M. which agrees by several research worker's data[24].

[Cd (L)(Anth)$_2$] complex: The diamagnetic properties showed as expected from their electronic configuration of Cd(II) d^{10} complex which did not display any peak in the visible region. The electronic spectrum of cadimum(II) complex shows the absorption peak at 342nm Table-3 which ascribed to the charge transfer, and which agrees with those complexes that have an octahedral structure [23], and the diamagnetic properties did not show any d-d transition,[24] .

6. Bacterial activities:

Schiff base ligand, Anthranilic acid and the new complexes were tested against various bacterial were given in chart-1.

The [Cr(L)(Anth)$_2$] shows negative against *E-coli* and *Bacillus*. The [Fe(L)(Anth)$_2$] shows negative result against *E-coli* and *Pseudomonas*. Complexes K [Co(L)(Anth$_2$] & K [Ni(L)(Anth$_2$] show good antibacterial activity against the 3-organisms except *E-coli*. The K[Cu(L)(Anth)$_2$] and K[Cd(L)(Anth)$_2$] show very good antibacterial activity against the four bacteria . Mostly the (ZI)mm of the prepared compounds was in the following order;

Metal complexes > AnthH > HL > DMSO

K[Cd(L)(Anth)$_2$] > K[Cu(L)(Anth)$_2$] > K [Ni(L)(Anth$_2$] >
K [Co(L)(Anth$_2$] >>[Cr(L)(Anth)$_2$] >[Fe(L)(Anth)$_2$]

It is obvious from the top datum that the (ZI) increased significantly on celates special the partial sharing of the (M^{+2} & M^{+3}) positive charge and electronic spectra of d^{10} of the metal ion with {N and O} atoms as donor groups or increased because of concept of cell permeability the lipid membrane that surround the cell surplus the vocal of only lipid soluble materials pump to which liposolubility is a significant operator that controls antimicrobial activity [29,30].

Table (1a): The physical properties for The starting materials and ligand HL

Compounds	Compound formula	Molecular weight	M .p°C	Colour	Found (Calc.)				
					C	H	N	O	S
M-dop	$C_{10}H_{13}NO_4$	211.22	290	White	56.86	6.20	6.63	30.30	-------
4-DMAB	$C_9H_{11}NO$	149.19	72-75	White	72.46	7.43	9.39	10.72	-------
HL	$C_{19}H_{22}N_2O_4$	342.39	160-165	Brown	64.95 (66.65)	6.87 (6.48)	8.66 (8.18)	13.32	-------

Table (1b): The solubility of the HL in various Solvents

Compound	H_2O	DMF	DMSO	CH_3OH	C_2H_5OH	C_3H_6O	C_6H_6	CCl_4	$CHCl_3$	Petroleume ether
HL	-	+	+	+	+	+	-	-	-	-

(+) soluble & insoluble (–)

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Table (1c): Physical Properties of the Mixed [L- Metal-Anth] Complexes

Compound formula	M. wt Calc.	Colour	Yield %	M .p°c	Λ_m Ω^{-1} cm^2 mol^{-1}	Metal% theory (exp)
$[Cr(L)(Anth)_2]$ $C_{33}H_{33}CrN_4O_8$	665.48	Brown	80	250 Dec	13.8	7.81 (6.83)
$[Fe(L)(Anth)_2]$ $C_{33}H_{33}FeN_4O_8$	669.48	Brown	89	190	9.5	8.34 (8.70)
$K[Co(L)(Anth)_2]$ $C_{33}H_{33}CoKN_4O_8$	711.67	Brown	67	220	31.4	8.28 (8.24)
$K[Ni(L)(Anth)_2]$ $C_{33}H_{33}N_4NiO_8$	711.43	Brown	72	225	36.2	8.25 (8.79)
$K[Cu(L)(Anth)_2]$ $C_{33}H_{33}CuN_4O_8$	716.28	Brown	78	240 Dec	38.1	8.87 (9.24)
$K[Cd(L)(A.nth_2]$ $C_{33}H_{33}Cd N_4O_8$	765.15	Brown	91	260 Dec	37.9	14.69 (13.97)

M.wt = {Molecular Weight},Λm={Molar Conductivity},Dec ={decomposition },M .p=melting point

Table (2a): FTIR of the M-dop and HL

compounds	υ (OH) arom	υ $(N-H_2)$	υ(C=N-)	(COO) υ asy υ sym HL	υ(C=C) arom. Alph.	υ (C-N)	$-CH_3$	υ(C-H) arom. alph.
M-dop	3481 3421br	3223s	--------	1618 1402	1490 1257	1209	1375	3015 2956,2808
HL	3473	--------	1620	1593 1365	1449 1273	1249	1315	3039 2966

Table (2b): FT-IR of the L-Anthranilic acid

$(NH_2)_{asym,sym}$ Str	υ $(N-H_3^+)$	C=O Str (carbox.)	$\upsilon(-COO^-)_{asym.}$	$\upsilon(-COO^-)_{sym.}$	Δυ $(-COO^-)_{asmy-smy}$
3321 s 3240	3101s	1716	1662s	1485s	177

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Table (2c): FT-IR of the complexes

Comp.	υ (OH) arom	υ (NH) asy,sym in Anth	υ (C–H) +CH3 aliph.	υ(HC=N-)	υ C=C arom	(-COO) asy,sym	Δ υ (-COO) asym-sym	υ (M-N) for L_3&(Anth)	υ (M-O) for L3&(Anth)
$[Cr(L)(Anth)_2]$	3444	3371 3078	2943 2816	1612	1523	1577 1365s	212	568 540	459 414
$[Fe(L)(Anth)_2]$	3479vs	3363 3062	2981m 2816	1612	1523	1581 1373	208	567 532	462 412
K [Co($(Anth_2]$	3433	3305 3136	2939 2812	1612	1535	1593 1386	207	586 563	455 416
K $[Ni(L)(Anth_2]$	3429	3305 3217	2943 2804	1604	1543	1593 1381	202	567 532	466 412
$K[Cu(L)(Anth)_2]$	3433	3275 3236	2947 2808	1604	1527	1554 1327	227	563 536	462 424
$K[Cd(L)(Anth)_2]$	3433	3290 3136	2939 2804	1616	1535s	1589 1400	189	578 555	451 412

Table (3): Electronic spectral of the mixed [L- Metal-Anth] Complexes

Comp.	λ_{max} nm	υ' cm^{-1}	€ max $Mol^{-1}.L.cm^{-1}$	Assignments	μ_{eff} B.M
$[Cr(L)(Anth)_2]$	342 798	29239 12531	2444 13	Charge transfer ${}^4A_{2g}\rightarrow{}^4T_{1g}(P)$ v3	3.94
$[Fe(L)(Anth)_2]$	344 790	29069 12658	1797 41	Charge transfer ${}^6A_1g\rightarrow{}^4T_1g(4P)$ $υ_3$	5.09
K [Co($(Anth_2]$	344 430 734 790	29069 23255 13623 12658	1691 70 54	Charge transfer ${}^4T_1g\rightarrow{}^4T_1g(p)$ $υ_3$ ${}^4T_1g\rightarrow{}^4T_2g(F)$ $υ_1$	4.36
K $[Ni(L)(Anth_2]$	344 797	29069 12547	1700 16	C.T ${}^3A_2g(F)\rightarrow{}^3T_1g(p)$ $υ_3$	2.83
$K[Cu(L)(Anth)_2]$	344 696	29069 14367	1712 55	Charge transfer ${}^2Eg\rightarrow{}^2T_2g$	1.62
$K[Cd(L)(Anth)_2]$	342	29239	1874	C.T	Diamagnetic

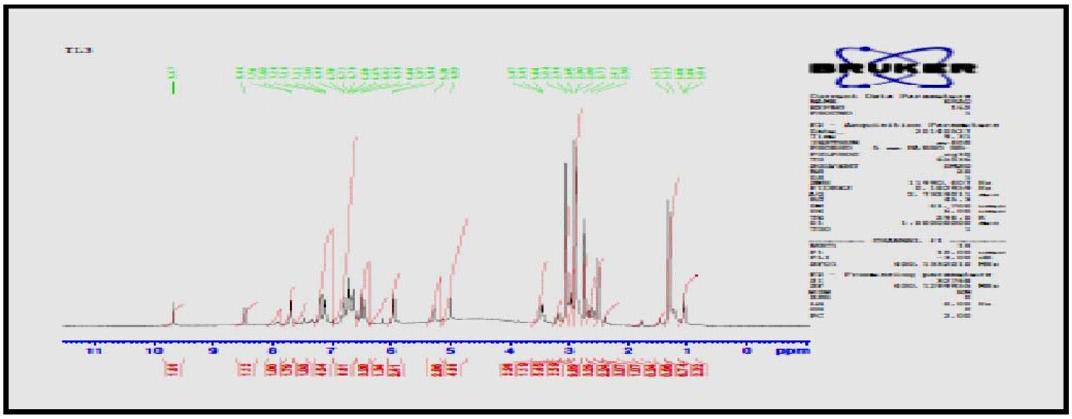

Figure (1): ^{1}H-NMR spectrum of the ligand (HL)

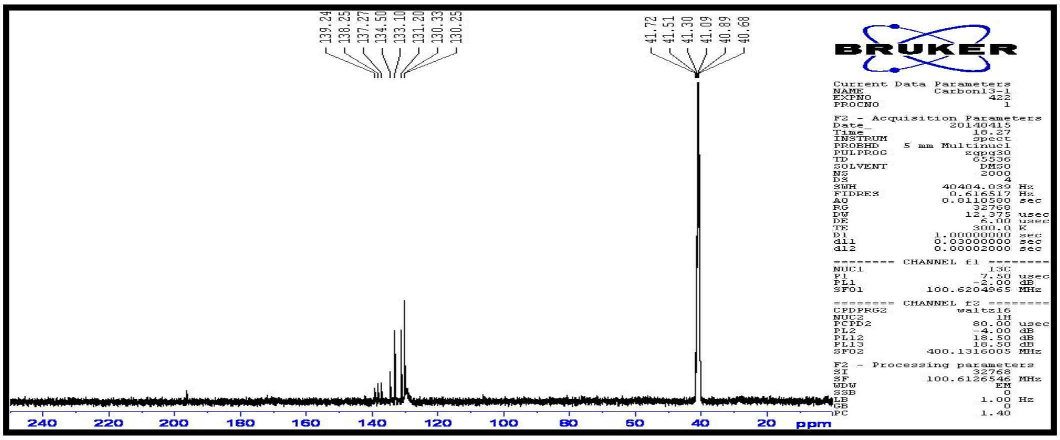

Figure (2): ^{13}C-NMR spectrum of the ligand (HL)

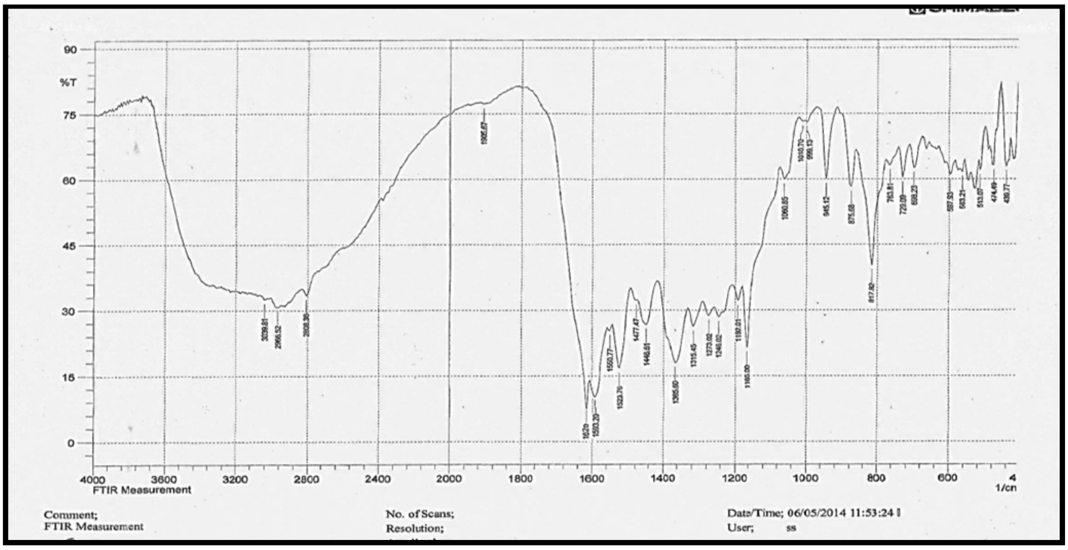

Figure (3): FT-IR of (HL)

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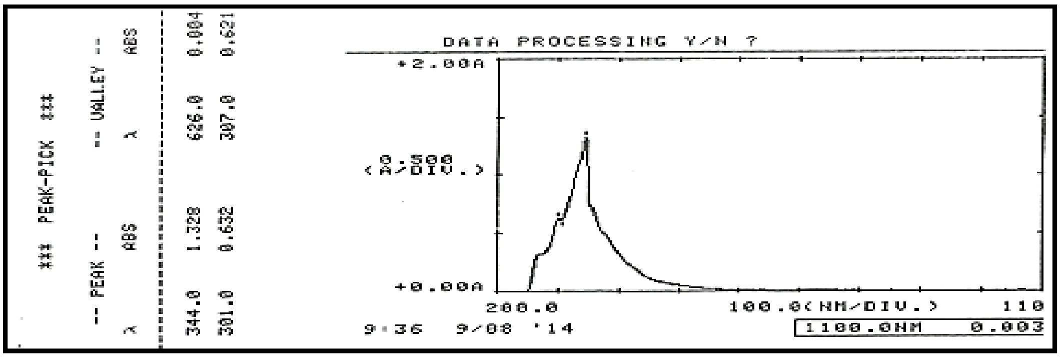

PEAK-PICK

-- PEAK --		-- VALLEY --	
λ	ABS	λ	ABS
344.0	1.328	626.0	0.004
301.0	0.632	307.0	0.621

Figure (4): Electronic spectrum of the (HL)

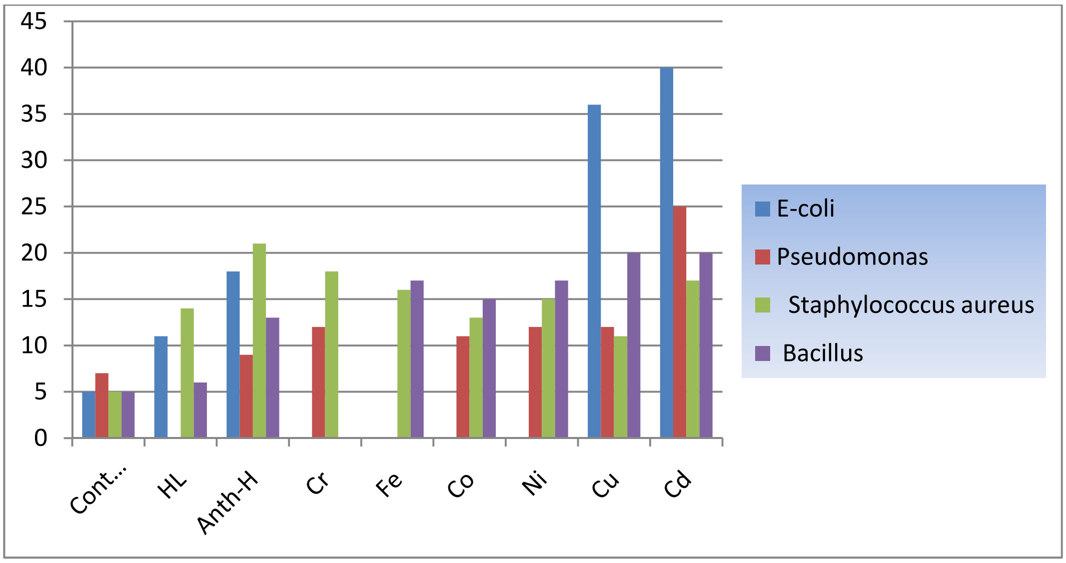

Figure (5): The (ZI) of mixed [L-Metal -Anth] complexes

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