Synthesis , Characterization and Biological Evaluation of the Cr(III), Mn(II), Fe(II), Fe(III), Co(II), Ni(II), Cu (II), Zn(II), Cd(II) and Hg(II) IonComplexes with Mixed Ligands (Quinolone Antibiotics and α-Aminonitrile Derivatives)

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Abstract

Quinolones L_1 (ciprofloxacin) are manufactured wide range anti-infection agents with great oral ingestion and magnificent bioavailability. Because of the concoction capacities found on their core (a carboxylic corrosive capacity at the 3-position) and much of the time an essential piperazinyl ring (or another N-heterocycle) at the 7-position and a carbonyl oxygen atom at the 4-position) quinolones bind metal particles framing buildings which can go about as bidentate. Bidentate ligands L_2=2-phenyl-2-(P-methoxy aniline) acetonitrile was set up by the response of Primiry amine with benzaldehyde, in nearness of potassium cyanide and acidic media . The metal edifices were portrayed by the miniaturized scale component examination (C.H.N), chloride contente , ^{1}H- NMR , ^{13}C-NMR , FT- IR and UV-Vis spectra, molar conductivity and in addition attractive vulnerability estimation. The coordination science and holding practices of the metal particles and ligands has been contem plated by spectra of the buildings on UV-vis and IR districts. As per the got information the possible coordination geometries of these buildings were proposed as octahedral. Some buildings were observed to be non - electrolyte others were observed to be weak electrolyte. The accompanying metal particle buildings were set up along side their recommended formulae based on the accompanying: The metal particle edifices of the ligands (L_1 and L_2) by build up an answer blend of [Cr(III), Mn(II), Fe(II), Fe(III), Co(II), Ni(II), Cu(II), Zn(II), Cd(II) and Hg (II)] particles separately,inabsolut ethanol with mixing gave the formulae : $[ML_1L_2Cl(H_2O)].2H_2O$ Where M:{ Mn^{2+},. Fe^{2+},Co^{2+},Ni^{2+} ,Cu^{2+},Zn^{2+},Cd^{2+} and Hg^{2+} }and $[ML_1L_2Cl(H_2O)]Cl.xH_2O$. Where M:{$Cr^{3+}$,$Fe^{3+}$},x:{2or3}

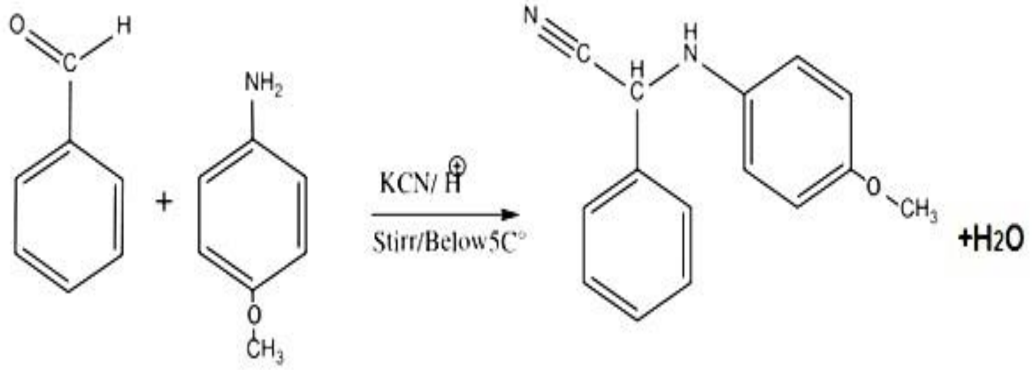
Keywords: quinolones (ciprofloxacin) 2-phenyl-2- (*P*-methoxy aniline) acetonitrile transition metal-ions.

Introduction

The utility of quinolone subsidiaries in territories of medication, nourishment, "impetus, color, materials , refineries and gadgets is well established."As an outcome, the amalgamation of quinolone center and its subordinates have been an appealing objective for the engineered natural scientific expert. Quinolones shape metal buildings because of their ability to tie metal ions [1]. In their metal buildings , the quinolone sn cango about as bidentate ligand , as unidentate ligand and as crossing over ligand. Much of the time, the quinolones are composed in a bidentate way, through one of the oxygen atom of deprotonated carboxylic of the ring and the ring of the carbonyl oxygen molecule. The nonspecific term "quinolone anti infection agents"alludes to the ring of manufactured anti-infection agents with bactericidal, the principal compound of the arrangement, was presented in treatment in the 1960 [2]. The clinical utilization of nalidixic corrosive was restricted by its limited range of movement. A few changes were made on the premise core keeping in mind the end goal to broaden the antibacterial rangent and to enhance the pharmaco kinetics properties, two of the seconsidered as being real: presentation of apiperazine moiety or another N-heterocyclic intheposition7 and presentation of a fluoride particle at the position 6[3].The new 4-quinolones , fluoroquinolones , have been found beginning in the1980[4].

Preparation of the Ligand

The ligand (L_2) was set up by taking after a formerly reported aside from changing of essential aromatic amine (P-methoxy aniline). Potassium cyanide (0.13g, 0.002mol) was dissolved in (4mL) of distilled water and cooled below 5°C. To this solution , benzaldehyde (0.212g, 0.002mol) in (25mL of 95% ethanol) was added. The mixture was mixed keeping up temperature below 5°C. glacial acidic acide (0.12g, 0.002mol) was included with constant mixing with keeping the temperature below 5°C, this was trailed by the expansion of P-methoxy aniline (0.476g, 0.002mole) in (10mL of 95% ethanol) and (5mL) of glacial acidic acide (cooled at 5°C) with constant blending in all around ventilated hood. The temperature was kept up at (15°C). By stirring the mixture about 2 hrs and was kept at room temperature for one day. The obtained pricipitate (long Purple needles), was washed with diluted HCl (0.2 mole) to remove any excess of potassium cyanide. The compound was recrystallized with 95% ethanol. The percentage yield of this procedure was (70.9%). The synthesis of the ligand was shown in reaction below :



Benzaldehyde *P*-methoxyaniline 2-phenyl-2-(*P*-methoxy aniline)acetonitrile(L_2)

Preparation of complexes(C_1-C_{10})

Preparation of (C_1) complex: a solution of (0.385g , 1mmol) of ciprofloxacin ligand (L_1) in (10mL) of absolut ethanol and (0.258gm, 1mmol) of 2-phenyl-2-(P-methoxy aniline) acetonitrile ligand in (10mL) of absolut ethanol was added drop wise to an answer of 1.0mmol of metal chloride (0.197g, 0.266g, 0.198g, 0.270g, 0.238g, 0.237g, 0.170g, 0.135g, 0.209g and 0.271g) for($CrCl_3.6H_2O$, $MnCl_2 4H_2O$, $FeCl_2.4H_2O$, $FeCl_3.6H_2O$, $CoCl_2.6H_2O$, $NiCl_2.6H_2O$, $CuCl_2.2H_2O$, $ZnCl_2$ and $HgCl_2$) separately, dissolved in (20mL) of total ethanol and refluxed with mixing under anhydrous surroundings utilizing Na_2SO_4 (anhydrous) for 24 hrs [7]. The acquired buildings were gathered after vanishing and the compounds were left in the desiccator to be dried under P_2O_5.

Results and Discussion

The significance of making α-aminonitrile compounds stand up from their versatility as starting materials for the synthesis of many compounds[8]. The configurations of prepared α-aminonitriles were identified by C. H. N. (Table 1), FT-IR (Table 2), ^{1}H-NMR and ^{13}C-NMR spectra (Table 3) and UV-visible (Table 4) techniques.

FTIR spectra of ciprofloxacin and α-aminonitrile ligands

The FT-IR spectral data of ciprofloxacin (L_1) and α-aminonitrile ligand (L_2) were shown in Table (2). L_1 ligand was showed the band at (1706 cm^{-1}) which was assigned to the υ(C= O) stretching vibration of the carboxylic group (L_1) and the most important stretching modes exhibited by (L_2) which was represented by α-amino and nitrile groups. The bands assigned to stretching vibration of aromatic and aliphatic (C-H) of L_1 and L_2 ligands appeared at (3000 - 3170) cm^{-1} and (2850 - 3000) cm^{-1} respectively [9]. The band related to υ(C≡ N) stretching vibration of the free ligand appeared at (2176cm^{-1})[10]. The band correlated to υ(N-H) stretching vibrations looked at (3348 cm^{-1})[11]. The band related to υ (N- H) bending vibration appeared at (1621) cm^{-1}[12-14].

FT-IR spectra of the complexes (C_1-C_{10})

The FT-IR spectral data of the complexes were shown in Table (2) and the spectrum of (C_8) was shown in Figure (1). The spectra of all the complexes exhibited characteristic bands of either coordinated water appeared at (3445-3506) cm^{-1} assigned to υ(OH) of the compexes (C_1-C_{10}) or cross section water showed up at (748-792) cm^{-1} assigned to ρw(H_2O) in all complexes [15-17]. New groups were presented up in the area (414-493) cm^{-1} and (501-585) cm^{-1} were assigned to υ(M- O) and υ(M-N), respectively [18]. The band which appeared at 3348 cm^{-1} was assigned to the υ(N-H) extending vibration to the (N-H) group of (L_2) was shifted in the spectra of the complexes (C_1- C_{10}) to (3357, 3358, 3361,3356,3363,3386,3357,3357,3357 and 3357)cm^{-1} respectively.This gave a sign that the ligand was collected with the metal ion through the nitrogen atom of α-amino group [19]. The band which appeared at (2176 cm^{-1}) was allocated to the extending vibration of υ(C≡ N) group of (L_2) which was moved in the spectra of complexes (C_1- C_4 and C_6 -C_{10}) to lower frequencies (2163, 2166, 2170, 2163, 2162, 2163, 2165, 2160 and 2166) cm^{-1} respectively which alludes to the linkage of (C≡ N) group from nitrogen atom. The range of complex (C_5) demonstrated an expanded move in υ(C≡N) extending vibration towards higher frequency as an effect of coordination with metal ion through the solitary combine electrons of nitrogen atom [20]. The declines of υ(C≡ N) extending vibration of metal-ion complexes(C_1-C_4 and C_6-C_{10}) were ascribed to metal dπ to ligand pπ∗ back-holding. Yet, the expansion of υ(C≡N) extending vibrationb of complex (C_5)

recognized to presence of π accepter ligands into complex (C_5) should decrease the back bonding of electrons from the metal into the nitrile ligand. The band at 1631 cm^{-1} which was consignedg to the δ(N-H) bending vibration of(N-H) group of (L_2) was shifted in the spectra of metal-ion complexes (C_1-C_{10}) to (1631, 1629, 1616, 1628, 1629, 1604, 1627, 1616 and 1627) cm^{-1} respectively , this provide more submission that theligand was coordinated with metal-ions through the nitrogen atom of α-aminogroup and thus supports the complexes formation [21]. The band at 1706 cm^{-1} which was assigned to the υ(C= O) stretching vibration of the carboxylic groupj of (L_1) as well ,ionic carboxy likcgroup have two absorption bands in the range of (1600- 1510) $cm^{-1}$and (1400- 1257) cm^{-1} which could bea ssigned to υ(C-O-C) asymmetric and symmetric which was removed in the spectra of stretching vibration separately[22]. The absorption band appear around (1485-1494) cm^{-1}, agreeing to the stretching vibrations of (C– C) and (C– N) bonds in quinolone ring (L_1). The coordination by two oxygen atoms from quinolone molecule (one oxygen from pyridine and the ether from the carboxylic group)[22].

^{1}H-NMR and ^{13}C-NMR spectra of the ligands and complexes

^{1}H-NMR

The two ligands (L_1 and L_2) were considered by ^{1}H-NMR and spectroscopic methods , in addition to sex complexes (C_2, C_5, C_7-C_{10}) using DMSO (**d6**) as shown in Table (3) and Figure (2). The ^{1}H-NMR spectra of the ligand (L_1) presented five peaks ; the first one looked at δ(1.42) ppm which was assigned to the (-C$\underline{H}_2$-), the second peak appeared at δ(2.22) ppm was assigned to the (=N$\underline{H}$) proton while the third peak appeared at δ(3.46) ppm which was agreed to the (-C$\underline{H}_2$-N=) [21]. The forward peak showed up at δ(7.45-7.55) ppm was ascribed to the fragrant protons and the last one showed up at δ(8.92) ppm assigned to the (- COOH) [22]. The ^{1}H- NMR spectra of the ligand (L_2) demonstrated five peaks ; The first one was showed up at δ(2.51) ppm which was related to the (- O-CH_3) protons, the second peak showed up δ(2.77) ppm was assigned to the dissolvable peak of (DMSO), the third peak showed up at δ(3.62) ppm was compares to the (- NH) proton , the fourth one was showed up at δ(5.35) ppm which was credited to the (- CH-C≡N) proton and the last peak showed up at δ(6.82- 8.00) ppm which was compared to the fragrant protons [23]. The ^{1}H- NMR spectra of the rest of the complexes (C_2, C_5, C_7-C_{10}) were likewise like that of the ligands, the main distinction was that the standard of (N-H) of the ligand was move in these compounds by (0.52, 0.36 and 0.36) ppm respectively this provided a sign for organizations arrangement.

^{13}C-NMR spectra

The two ligands (L_1 and L_2) were described by ^{13}C-NMR spectroscopic techniques, in addition to sex compounds (C_2, C_5, C_7-C_{10}) utilizing DMSO (**d6**) as appeared in Table (3) and Figure (3). The ^{13}C- NMR range of (L_1) demonstrated seven peaks ; the principal peak showed up at δ(7.7) ppm which was related to the (- $\underline{C}H_2$- $\underline{C}H_2$-). The second peak showed up at δ(35.8) ppm was compared to the (= $\underline{C}$H -N=) carbon, the third peak showed up at δ(45.8) ppm was allocated to the (- $\underline{C}H_2$- NH-) carbon , the forward peak showed up at δ(51.3) ppm was assigned to the (- $\underline{C}H_2$-N=) carbon , the fifth peak showed up at δ(166.2) ppm which was appointed to the (- $\underline{C}$OOH) carbon , the 6th peak showed up at δ(176.4) ppm was assigned out to the (- $\underline{C}$=O) carbon , and the last peak showed up at δ (102-115) ppm was alloted to the sweet-smelling carbon molecules [21]. The ^{13}C-NMR range of the ligand (L_2) demonstrated three character tops; the firest principal top was showed up at δ(38.04) ppm which was compared to the dissolvable peak 0f (DMSO), the second peak was showed up at δ(52.20)ppm which related to the (- C̲H-C≡N) carbon and the last peak showed up at δ (115.37) ppm was appointed to the (- CH- C̲≡N) carbon particle [22]. The ^{13}C-NMR spectra of the complexes (C_2, C_5, C_7-C_{10}) were like that of the ligands, the main contrast was that the standard of (- C̲H -C≡N) carbon of the (L_2) ligand (was moved in these complexes by (0.83, 8.41, 3.38, 0. 29, 1.23 and 3.7) ppm respectively. The standard of (- CH- C̲≡N) carbon was moved in these compounds by (1.99, 1.75, 4.66, 1.4, 1.26 and 1.4 2) ppm and the standard of (- C̲OOH) of (L_1) was moved in these complexes by (1.99, 1.75, 4.66, 1.40, 1.26 and 1.40) and the standard of (- C̲=O) of a similar ligand was moved in these complexes by (3.00, 0.74, 2.41, 1.75, 0.21 and1.96) respectively, this gave a sign for buildings arrangement[23].

Electronic spectra(UV-Vis.)

The electronic absorption data of the ligands (L_1 and L_2) and their metal ion complexes were recorded in ethanol at room temperature was shown in Table(5), while some of their spectra were shown in Figures (4, 5 and 6). The spectra of the two ligands was shown a high intensity bands looked in the region (48543 and 41666)cm^{-1} respectively, assigned to $\pi\rightarrow\pi^*$ of conjugated system [24]. Low intensity bands appeared in near UV-visible region (31645 and 34364) cm^{-1} correspondingly, were given to $n\rightarrow\pi^*$ transition, the concentration and positions of these bands be influenced by the structure of molecules and the nature of the used solvent [25]. The electronic spectra of the complexes exhibited new bands, with intensities and places of these bands are mostly dependent on the ligand field effects, stereochemistry of complexes and electron shape of the metal ions [26].

Thermal analysis of complexes

Warm decay of the comlexes (C_1-C_3 and C_5-C_9) takes after TG strategies, warming extent (30-450)°C and warming rate (10 °C/min). The warm decay information for all comlexes have three stages where given in Table (5) and their thermographs of Zn(II) complex (C_8) was appeared in Figure (7). The accompanying results were acquired:

1.The principal organize which occurred at temperature scope of (50-150)°C. incorporate leaving the lattice water atoms and counter chloride particles [27,28].

2.The accompanying stage incorporate leaving the organized water particles and take off the chloride particles at temperatures relying upon security energies came about because of coordination of ligands [29,30].

3.The last phase of warm decay of comlexes gave metal oxides as last live contingent upon the sort of metal particle and on (M-L) proclivity [31,32].

In vetro Antibacterial Activity

The antibacterial exercises of all compounds were divided against test microbes in particular; *Staphylococcuaurouss, Bacillus* subtilis (Gram+), *Escherichia* and *Pseudomonas* aerugin (Gram-). Agar , (well-dispersion strategy) used to decide the movement [33]. Borer of 0.6 mm distance across was utilized, the convergence of all compounds was (10^{-3}M) utilizing (DMSO) as a dissolvable and was utilized as a control Gram negative while ciprofloxacin was utilized as a control Gram positive. The dissolvable (DMSO) demonstrated no action against the tried microscopic organisms, while some of arranged compounds indicated great results [34]. Table (6) demonstrates the hindrance zones of the dissolvable and the arranged buildings. The hindrance zones were measured in (mm) and contrasted with the restraint zone of expansive range anti-infection. All edifices demonstrated no movement against the gram negative bacterium (*Escherichia coli*), which can bring about ailment, for instance, enter toxigenic strains deliver a poison in the gut, coming about regularly in looseness of the bowels [35]. The complexes (C_2 and C_4) demonstrated most elevated movement against Pseudomonas and *Staphylococcus* aureus. This bacterium is known for its imperviousness to the greater part of the created anti-infection agents and is known to be the real instance of numerous medical problems and contaminations [36]. Numerous elements were accounted for to control the organic exercises of metal buildings [37]. Sort of ligand, kind of metal, charge of complex, the move arrangement, design of metal particle and geometry of metal complex are affected on organic activity[38].

The nomenclature of suggested structures of the complexes

The suggested structures of the prepared complexes were confirmed by their elemental analysis (C.H.N.), thermal analysis, infrared,^{1}H-NMR and ^{13}C-NMR spectra, UV-Vis.spectroscopy and by molar conductance values. According to the observation obtained the structures of the metal ion complexes are suggested as illustrated in Figures (8, 9 and 10) below:

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Table [1]: Elemental micro analysis and some physical properties of the ligands (L1 and L2) and their prepared complexes

COMP.	FORMULA M.W.T	YIELD %	COLOR	M.P (°C)	C % CAL (FOUND)	H % CAL (FOUN)	CAL (FOUN)	M % CAL (FOUND)	CHLORINE %
L_1	$C_{17}H_{21}FClN_3O_4$ (385.8)	-----	Light White	255-257	52.87 (52.20)	5.44 (5.13)	10.88 (10.07)	------	9.18 (8.65)
L_2	$C_{15}H_{14}N_2O$ (238)	77.6	Leaden	79-83	75.63 (74.88)	5.88 (5.27)	11.76 (11.08)	------	------
C_1	$Cr[L_1L_2Cl(H_2O)]Cl.3H_2O$ (764.7)	86.2	Green	317 Dec.	50.21 (49.48)	4.05 (3.66)	9.15 (7.98)	6.66 (5.19)	9.27 (7.87)
C_2	$[MnL_1L_2Cl(H_2O)]\ 2H_2O$ (749.2)	71.7	Pall Yellow	350-352	51.25 (49.76)	3.87 (4.03)	9.34 (7.90)	7.32 (6.10)	9.46 (7.90)
C_3	$[FeL_1L_2Cl(H_2O)]\ 2H_2O$ (714.6)	65.8	Yellow	319 Dec.	53.73 (50.76)	4.05 (3.37)	9.56 (8.84)	7.62 (6.78)	4.84 (3.09)
C_4	$[FeL_1L_2Cl(H_2O)]Cl.2H_2O$ (750.1)	75.2	Yellow	329-332	51.19 (48.75)	3.86 (3.59)	9.33 (7.43)	7.43 (6.1)	9.45 (8.11)
C_5	$[CoL_1L_2Cl(H_2O)]\ 2H_2O$ (717.7)	82.4	Blue	290 Dec.	53.50 (50.68)	4.04 (3.85)	9.75 (8.03)	8.20 (7.26)	4.93 (93.38)
C_6	$[NiL_1L_2Cl(H_2O)]\ 2H_2O$ (717.4)	73.6	Metallic Bronze	310-312	53.52 (51.73)	4.04 (4.13)	9.75 (8.10)	8.16 (6.56)	4.93 (3.57)
C_7	$[CuL_1L_2(H_2O)_2]\ Cl.1.5H_2O$ (695.3)	73.4	Gray	285 Dec.	55.23 (54.91)	4.31 (3.92)	10.06 (8.76)	9.13 (7.1)	5.09 4.78
C_8	$[ZnL_1L_2Cl(H_2O)]\ 2H_2O$ (724.2)	68.9	White	294 Dec.	53.02 (50.42)	4.00 (4.58)	9.66 (8.47)	9.03 (7.32)	4.88 (3.75)
C_9	$[CdL_1L_2Cl(H_2O)].2H_2O$ (771.2)	79.1	White	288 Dec.	49.79 (46.76)	3.76 (4.39)	9.07 (7.16)	14.57 (13.03)	4.59 (3.10)
C_{10}	$[HgL_1L_2Cl(H_2O)]\ 2H_2O$ (894.8)	67.1	White	308 Dec.	43.82 (---)	4.56 (---)	7.98 (---)	22.82 (---)	8.02 (---)

Dec.=Decomposition

Table(2): Characteristic infrared absorption bands of ligands (L_1 and L_2) andtheir metal ions com plexes

Comp.	υ(N−H) cm^{-1}	υ(C≡N) cm^{-1}	υ(C=O) cm^{-1}	δN−H cm^{-1}	υ(C-O-C) (asy)cm^{-1}	υ(C-O-C) (sy)cm^{-1}	υ(M-O) cm^{-1}	υ(M-N) cm^{-1}
L_1	3378 w.sh	------	1707 v.sh	1625 s.sh	------	------	------	------
L_2	3348 w.sh	2176 s.sh	------	1621	------	------	------	------
C_1	3357 m.sh	2164 s.sh	------	1631 m.sh	1520 w.sh	1300 s.sh	585 w	418 w
C_2	3358 w.sh	2167 m.sh	------	1629 s.sh	1557 m.sh	1300 s.sh	501 w	441 w
C_3	3361 m.br	2170 m.sh	------	1616 w.br	1514 w.sh	1292 s.sh	555 w	443 w
C_4	3356 m.br	2163 m.sh	------	1628 v.sh	1504 m.sh	1247 m.sh	547 w	483 w
C_5	3363 m.br	2204 w.br	------	1629 v.sh	1571 s.sh	1305 m.sh	572 w	420 w
C_6	3386 m.br	2162 w.sh	------	1622 s.sh	1504 s.sh	1247 s.sh	547 w	493 w
C_7	3357 w.sh	2165 m.sh	------	1604 m.sh	1562 m.sh	1302 s.sh	574 w	420 w
C8	3357 m.sh	2165 v.s.sh	------	1627 m.sh	1573 w.sh	1402 w.sh	514 w	414 w
C_9	3357 m.sh	2160 m.sh	------	1616 s.sh	1566 w.sh	1271 s.sh	541 w	470 w
C_{10}	3357 m.sh	2166 s.sh	1712 m.sh	1627 m.sh	-----	-----	557 w	458 w

where: s=strong , m=medium , w=weak , v=very, br=broad , sh=sharp

Table (3): ^{1}H-NMR and ^{13}C-NMR of the ligands (L1 and L2) and som of their metal ion complexes

Comp.	Formula	Groups	Chemical Shifts δ(ppm)	Groups	Chemical Shifts δ(ppm)
L_1	$C_{17}H_{21}FClN_3O_4$	(-C$\underline{H}_2$-) (=N$\underline{H}$) (-C$\underline{H}_2$-N=) (Ar-$\underline{H}$) (-COO$\underline{H}$)	1.42 2.22 3.46 7.45-7.55 8.92	(-CH_2- CH_2-) (-$\underline{C}$-H-N=) (- $\underline{C}H_2$-NH-) (- $\underline{C}H_2$-N=) (- COOH) (-C=O)	7.7 35.8 45.8 51.3 166.2 176.4
L_2	$C_{15}H_{14}N_2O$	(-O-C$\underline{H}_3$) (=N$\underline{H}$) (=C$\underline{H}$- ≡N)	2.51 3.62 5.35	(- $\underline{C}$H- ≡N) (-CH-$\underline{C}$≡ N)	52.20 115.37
C_2	$[MnL_1L_2Cl(H_2O)]Cl.2H_2O$	(-N$\underline{H}$) (=C$\underline{H}$-C≡N) (Ar-$\underline{H}$)	3.98 5.59 7.62-7.93	(- $\underline{C}$H-C ≡N) (-CH-$\underline{C}$ ≡N) (-$\underline{C}$OOH)	60.01 120.06 167.46
C_5	$[CoL_1L_2Cl(H_2O)]\ 2H_2O$	(-N$\underline{H}$) (=C$\underline{H}$-C≡N) (Ar-$\underline{H}$)	3.55 5.87 6.90-7.89	(- $\underline{C}$H-C ≡N) (-CH-$\underline{C}$ ≡N) (-$\underline{C}$OOH)	57.95 118.91 168.19
C_7	$[CuL_1L_2(H_2O)2]\ 1,5H_2O$	(=N$\underline{H}$) (=C$\underline{H}$- ≡N) (Ar-$\underline{H}$)	3.91 5.80 6.81-7.92	(- CH- ≡N) (-CH-$\underline{C}$≡N) (- $\underline{C}$OOH)	58.49 115.98 167.60
C_8	$[ZnL_1L_2Cl(H_2O)]\ 2H_2O$	(=N$\underline{H}$) (=C$\underline{H}$-C≡N) (Ar-$\underline{H}$)	3.83 5.71 6.81-7.85	(- $\underline{C}$H-C ≡N) (-CH-$\underline{C}$ ≡N) (-$\underline{C}$OOH)	55.40 119.40 161.54
C_9	$[CdL_1L_2Cl(H_2O)]\ 2H_2O$	(=N$\underline{H}$) (=C$\underline{H}$-C≡N) (Ar-$\underline{H}$)	3.84 5.71 6.98-7.92	(- $\underline{C}$H- ≡N) (-CH-$\underline{C}$≡N) (- $\underline{C}$OOH)	50.37 119.38 167,95
C_{10}	$[HgL_1L_2Cl(H_2O)]Cl.2H_2O$	(-N$\underline{H}$) (=C$\underline{H}$-C≡N) (Ar-$\underline{H}$)	3.79 5.71 6.88-7.92	(- $\underline{C}$H- ≡N) (-CH-$\underline{C}$≡N) (- $\underline{C}$OOH)	55.08 119.39 164.78

Table (4): Electronic spectra, parameter, molar conductance, magnetic susceptibility and suggested stereo chemical of the ligands and their metal ion complexes

Comp.	Wavlength	Wave no. cm^{-1}	Assignment	Molar Cond. $\Omega^{-1}\ cm^{2}\ mol^{-1}$	B. M	B^{-} cm-1	β	Dq/B^{-}	$15B^{-}$ cm-1	Geometry Suggested
L_1	316 280	31645 48543	$n\rightarrow\pi^{*}$ $\pi\rightarrow\pi^{*}$	-------	----	----	----	----	----	---------
L_2	291 240	34364 41666	$n\rightarrow\pi^{*}$ $\pi\rightarrow\pi^{*}$	------	------	----	----	----	----	---------
C_1	---- 600 448	16111* 16666 22321	$^{4}A_{2g}\rightarrow{}^{4}T_{2g}$ $^{4}A_{2g}\rightarrow{}^{4}T_{1g(F)}$ $^{4}A_{2g}\rightarrow{}^{4}T_{1g(P)}$	50.9	3.91	771	0.840	1.25	11566	Octahedral
C_2	506	19762	$^{6}A_{1g}\rightarrow{}^{4}T_{1g}$	22.3	1.41	----	----	----	----	Octahedral
C_3	503	19880	$^{5}T_{2g}\rightarrow{}^{5}E_{g}$	18	2.34	----	----	----	----	Octahedral
C_4	758 645	13192 15503	$^{6}A_{1g}\rightarrow{}^{6}T_{1g(G)}$ $^{6}A_{1g}\rightarrow{}^{4}A_{1g}+{}^{4}E_{g}$	29.1	5.72	----	----	----	----	Octahedral
C_5	659 ---- 490	15174 15501* 20408	$^{4}T_{1g(F)}\rightarrow{}^{4}T_{2g(F)}$ $^{4}T_{1g(F)}\rightarrow{}^{4}A_{2g}$ $^{4}T_{1g(F)}\rightarrow{}^{4}T_{1g(P)}$	23.3	4.68	641	0.660	1.24	9613	Octahedral
C_6	778 591 224	12853 16920 44642	$^{3}A_{2g}\rightarrow{}^{3}T_{1g}$ $^{3}A_{2g}\rightarrow{}^{3}T_{1g(F)}$ $^{3}A_{2g}\rightarrow{}^{3}T_{1g(p}$	23.5		----	----	----	----	Octahedral
C_7	503	19880	$^{2}E_{g}\rightarrow{}^{2}T_{2g}$	35.8	1.97	----	----	----	----	Distorted Octahedral
C_8	327 288	30581 34722	$n\rightarrow\pi^{*}$	21.2	-----	----	----	----	----	Octahedral
C_9	321 284	31152 35211	$n\rightarrow\pi^{*}$	23.1	-----	----	----	----	----	Octahedral
C_{10}	310 281	32258 35587	$n\rightarrow\pi^{*}$	13.6	-----	----	----	----	----	Octahedral

* calculated value

Table (5):Thermal decomposition data for some metal ion complexes

Comp.	Molecular formula	step	Temp. rang of the Decomposition C°	Suggested Formula of resultant compound	Mass loss%	
					Cal.	found
C_1	$[CrL_1L_2Cl(H_2O)]Cl.3H_2O$	1	80-170	Cr $[L_1L_2(H_2O)(Cl)]Cl$	7.04	5.86
		2	170-210	$Cr[L_1L_2]$	11.6	10.4
		3	210-375	Cr_2O_3	76.8	74.5
C_2	$[MnL_1L_2Cl(H_2O)]Cl.2H_2O$	1	94-152	Mn $[L_1L_2(H_2O)(Cl)]C$	4.58	4.04
		2	152-233	$lMn[L_1L_2]$	11.3	10.2
		3	233-397	MnO	74.9	73.5
C_3	$[FeL_1L_2Cl(H_2O)]\ 2H_2O$	1	50-105	$[FeL_1L_2Cl(H_2O)]$	4.92	3.78
		2	105-175	$[FeL_1L_2]$	7.3	6.14
		3	175-380	FeO	76.7	73.7
C_5	$[CoL_1L_2Cl(H_2O)]\ 2H_2O$	1	60 -140	Co $[L_1L_2(H_2O)(Cl)]$	4.9	3.89
		2	140 -195	$[CoL_1L_2]$	7.27	6.24
		3	195 -360	CoO	80.1	76.8
C_6	$[NiL_1L_2Cl(H_2O)]\ 2H_2O$	1	73 -127	Ni $[L_1L_2(H_2O)(Cl)]$	4.9	4.3
		2	127 -186	$Ni[L_1L_2]$	7.28	6.8
		3	186 -350	NiO	80.2	79.10
C_7	$[CuL_1L_2(H_2O)_2]\ 1,5H_2O$	1	54 -100	Cu $[L_1L_2(H_2O)_2]$	3.95	3.76
		2	100 -153	$Cu[L_1L_2]$	5.27	4.89
		3	153 -388	CuO	86.2	85.1
C_8	$[ZnL_1L_2Cl(H_2O)]\ 2H_2O$	1	57 -142	$Zn[L_1L_2(H_2O)_2]Cl$	4.85	3.76
		2	142 -165	$Zn[L_1L_2]$	7.15	6.01
		3	165-376	ZnO	79.4	78.6
C_9	$[CdL_1L_2Cl(H_2O)]\ 2H_2O$	1	55-130	Cd $[L_1L_2(H_2O)(Cl)]$	4.56	3.74
		2	130-215	$Cd[L_1L_2]$	6.72	6.3
		3	215-380	CdO	74.7	72.8

Table (6):Inhibition zonesbmeasuredhin (mm) of DMSO, ciprofloxacin and metal ion complexes

Compound	Inhibition zone (mm) *Escherichia coli*	Inhibition zone (mm)*Pseudom*	Inhibition zone (mm)*Staphyl*	Inhibition zone (mm) *Streptococci*
DMSO	----	----	----	----
L_1(ciprofloxacin)	18.50	23.70	18.15	12.23
C1	12.00	35.00	7.00	14.00
C2	8.00	44.00	21.00	18.80
C3	11.00	25.10	6.00	13.00
C4	21.00	40.70	21.00	15.00
C5	22.00	29.40	22.50	18.00
C6	14.00	12.00	5.80	12.10
C7	12.00	14.00	23.00	13.00
C8	11.50	10.50	6.02	13.70
C9	10.00	11.00	22.50	15.20

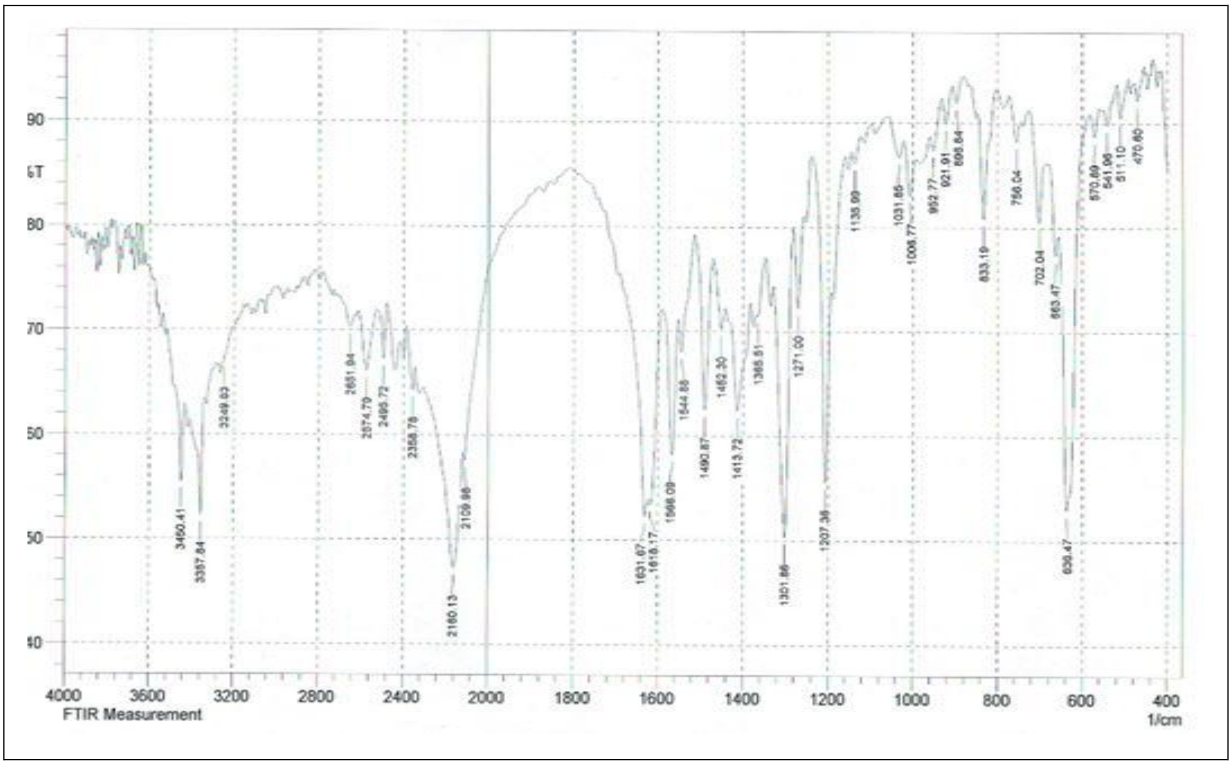
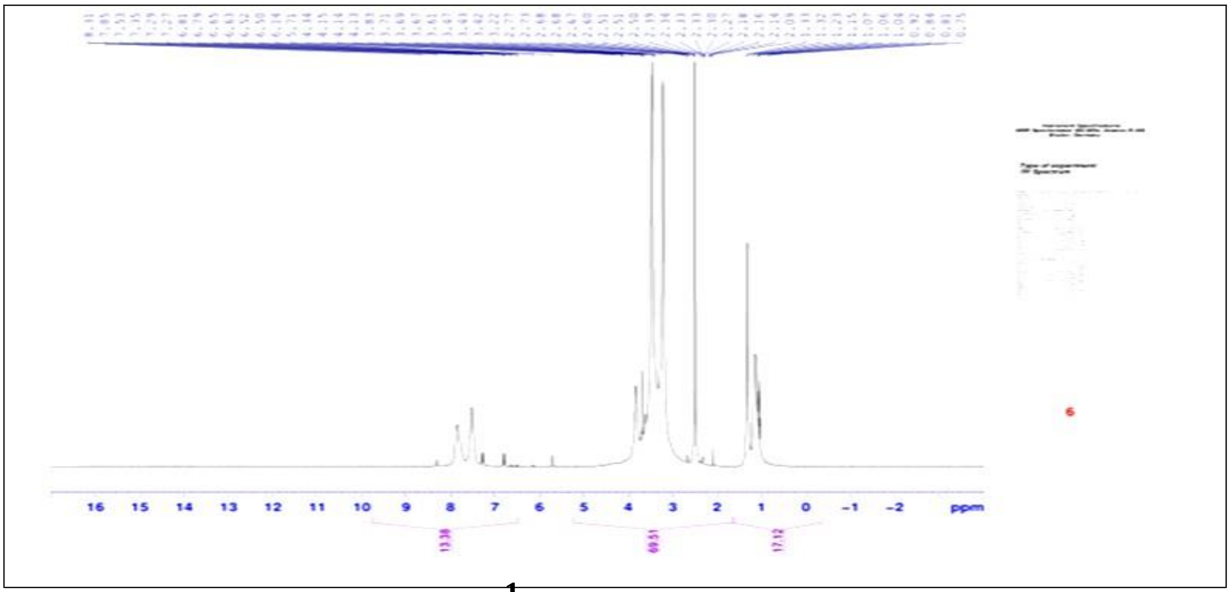
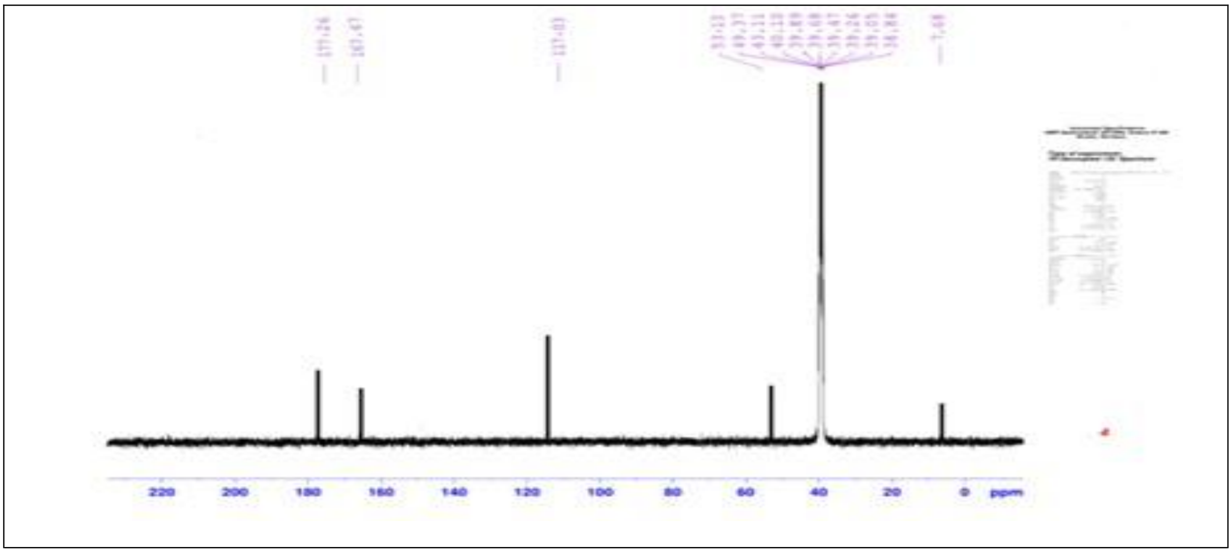


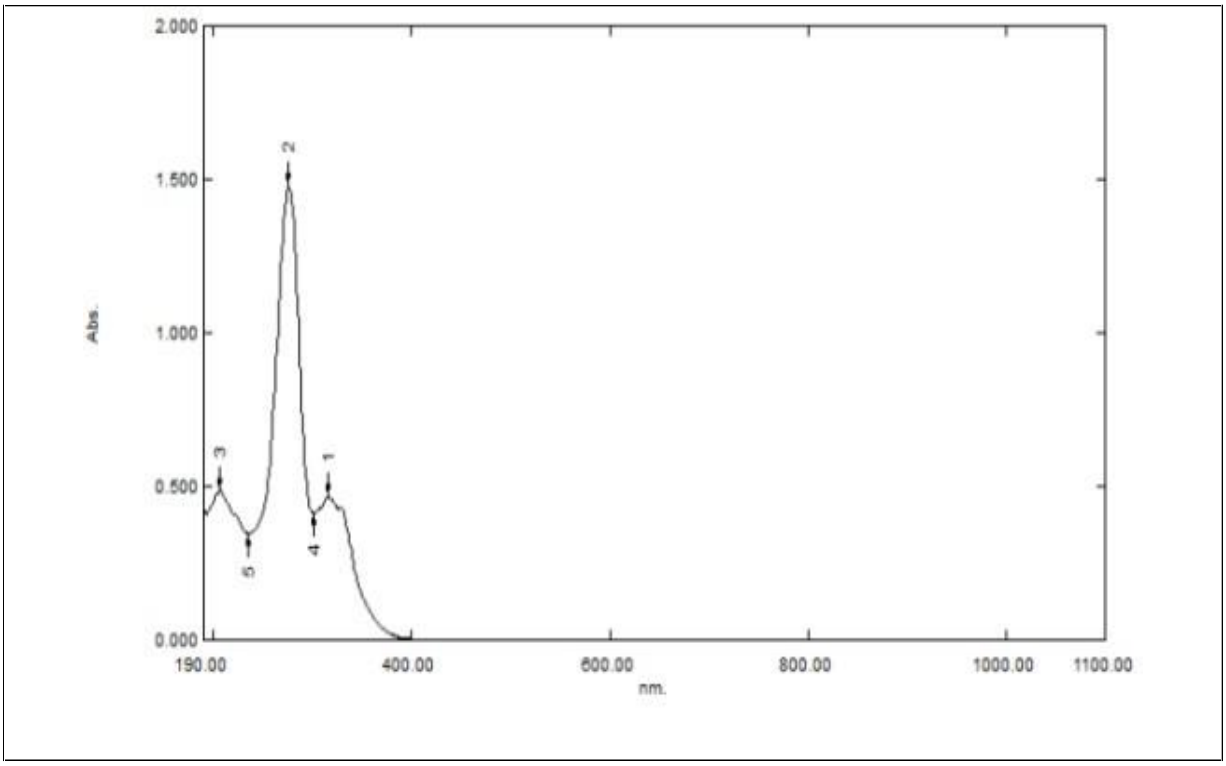
Figure (1): FT-IR spectrum of C_8



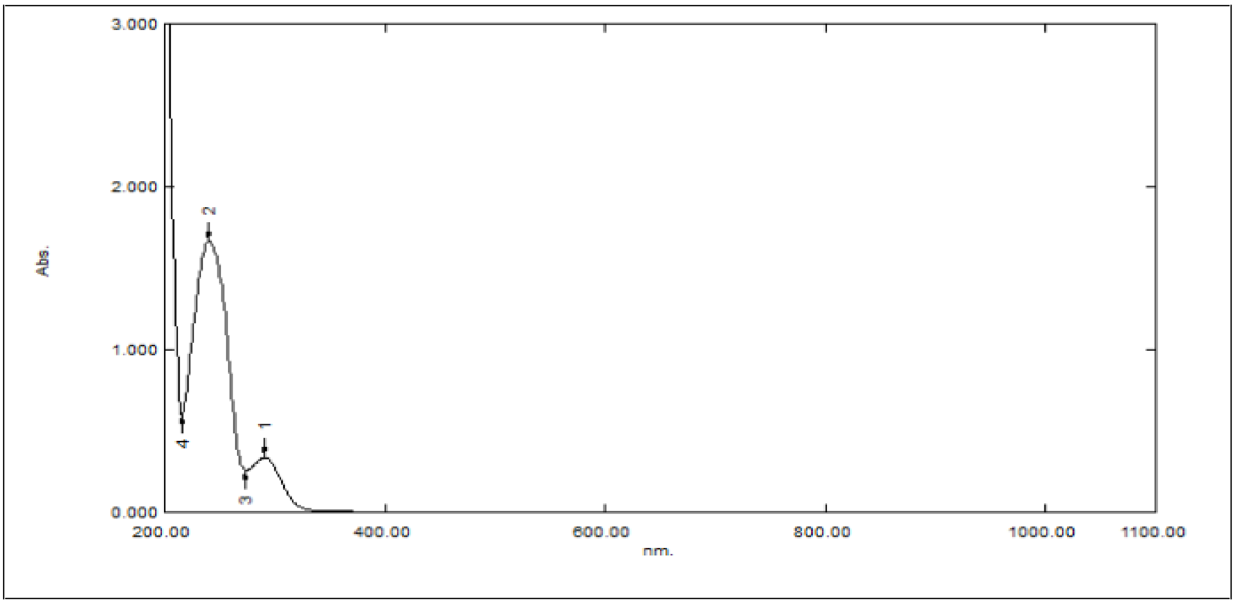
Figure(2): ^{1}H-NMR spectrum of C_8



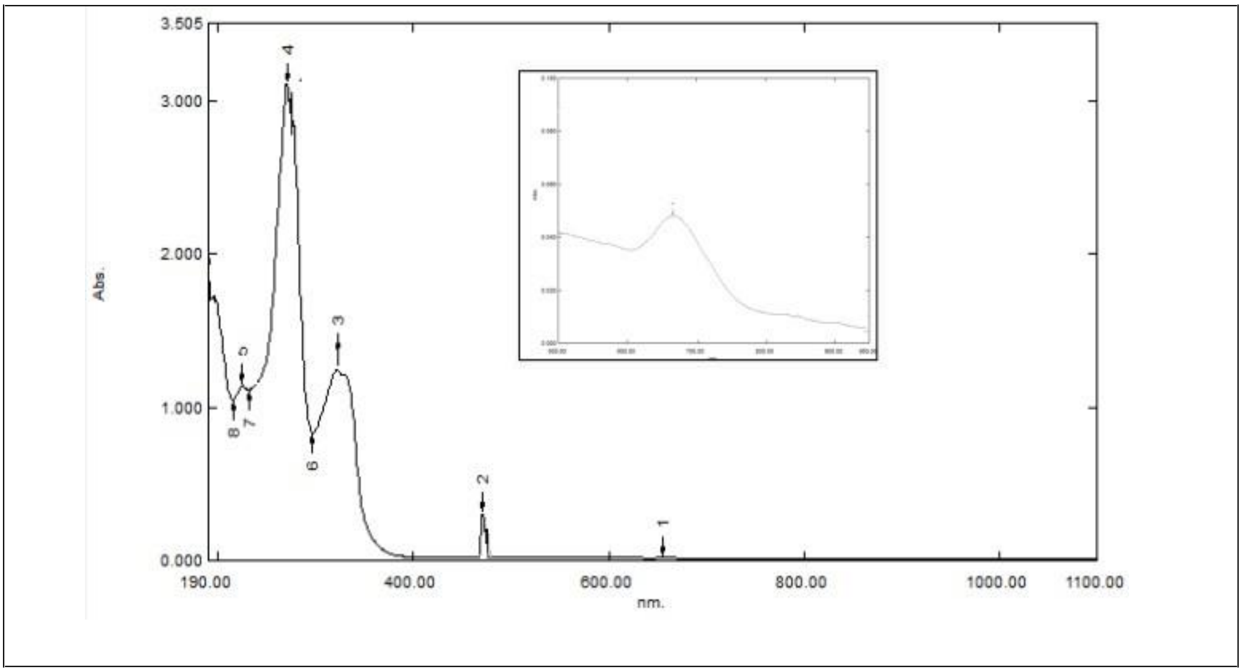
Figure(3): ^{13} **C-NMR spectrum of** C_8



Figure(4): UV-Visible Spectrum of L_1



Figure(5): UV- Visible Spectrum of L_2



Figure(6): UV-Visible Spectrum of C_1

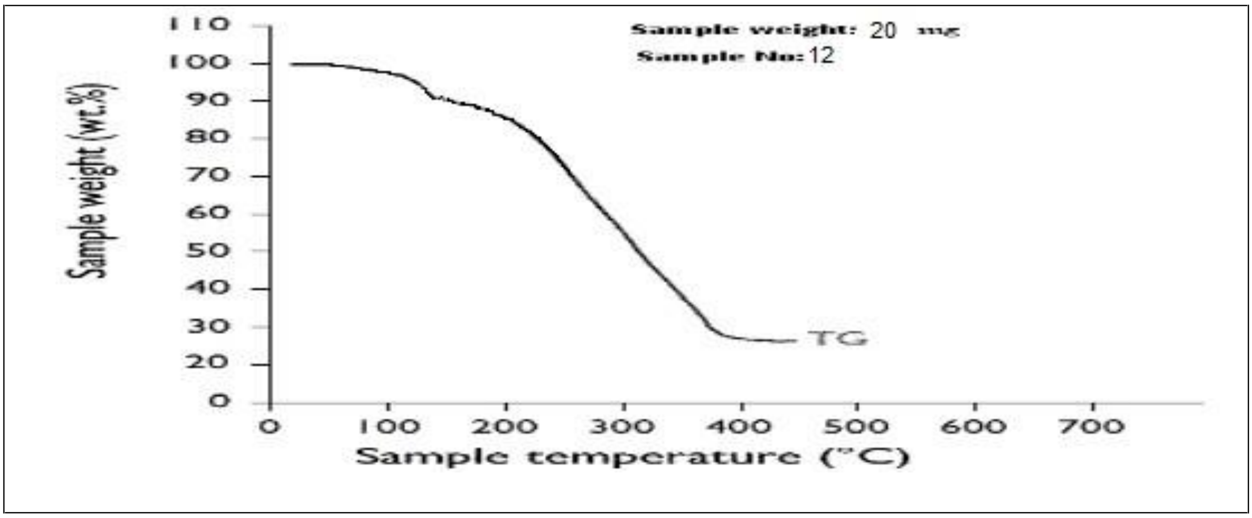
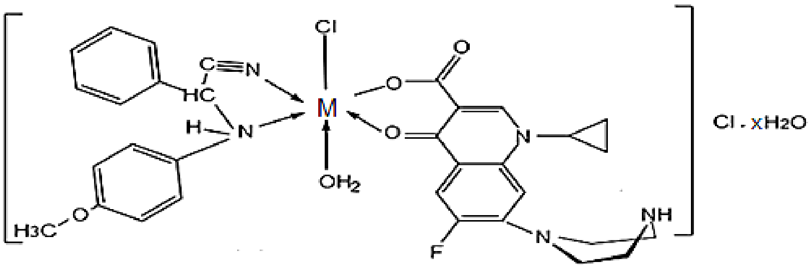


Figure (7): Thermograph of C_8



Where: M {Cr^{3+} or Fe^{3+}} and x{2 or 3}

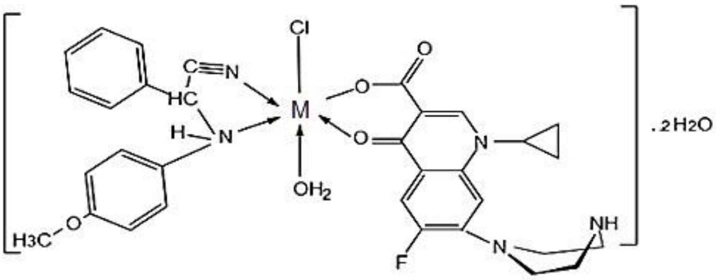
$[CrL_1L_2Cl(H_2O)]Cl.3H_2O$

[chloro mono aqua ciprofloxacin {2-phenyl -2-(*P*- methoxy anilino) acetonitrile}chrome (III)] chloride (3)hydrate

$[FeL_1L_2Cl(H_2O)]Cl.2H_2O$

[chloro mono aqua ciprofloxacin {2-phenyl-2-(*P*- methoxyanilino)acetonitrile} iron(III)] chloride dihydrate

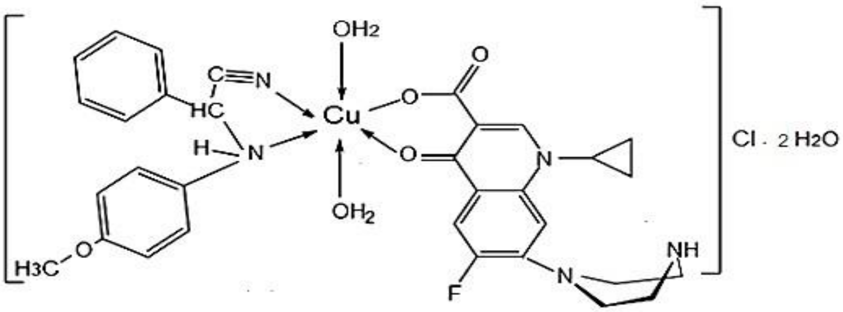
Figure (8): Suggested the structures of C_1 and C_4 complexes



$ML_1L_2Cl(H_2O)].2H_2O$

Where: M {Mn^{2+}, Fe^{2+}, Co^{2+}, Ni^{2+}, Zn^{2+}, Cd^{2+} and Hg^{2+}} [chloro mono aqua ciprofloxacin {2-phenyl-2-(*P* -methoxyaniline)acetonitrile } metal (II)] dehydrate

Figure (9): Suggested structure of C_2, C_3, C_5, C_6, C_8 and C_{10} complexes



$[CuL_1L_3(H_2O)_2]Cl.\ 2H_2O$

[diaqua ciprofloxacin {2-phenyl -2-(*P*- methoxyaniline) acetonitrile } copper(II)]Chloride dihydrate

Figure(10): Suggested structure of C_7 complex

تحضير وتشخيص و تقييم الفعالية البيولوجي من الكروم (III) و المنغنيز (II) والحديد (II) والحديد (III) والكوبلت (II) والنيكل (II) والنحاس (II) والزنك (II) والكادميوم (II) والزئبق (II) ايون معقدات من مزيج الليكندات (سبروفلوكساسين ومشتقات الفا امينو نتريل)

محمد حسين عباس العامري
سعد خضر محمد
قسم الكيمياء/ كلية العلوم /جامعة بغداد

استلم في:27/كانون الأول/2016، قبل في:15 /اذار/2016

الخلاصة

الكينولين (سيبروفلوكساسين) هي المضادات الحيوية الاصطناعية واسعة الطيف مع امتصاص عن طريق الفم جيدة والفعالية البيولوجية ممتازة . ونظرا إلى وظائف مادة كيميائية موجودة في النواة (وظيفة حمض الكربوكسيلية في موقف (3) وفي معظم الحالات حلقة الأساسية (piperazinyl) أو (N-heterocyclic) في موقع(7) وذرة أوكسجين مجموعة الكربونيل في موقع (4) وارتباط الكينولين بأيونات المعادن ثنائي السن . بروابط ثنائي السن 2-فينيل-2-/ تم إعداد (P- ميثوكسي الأنيلين) الأسيتو نتريل بواسطة تفاعل الأمينات الأولية مع بنزيلديهايد، في وجود سيانيد البوتاسيوم في الوسط الحمضية. شخصت المعقدات المحضرة كميا ونوعيا باستخدام تحليل العناصر C.H.N ,طيف الأشعة تحت الحمراء , طيف الاشعة فوق البنفسجية-المرئية الرنين النووي المغناطيسي ,الامتصاص الذري ,الخاصية المغناطيسية, التحاليل الحرارية وقياس التوصيلية .وفقا للبيانات التي تم الحصول عليها اقترح الشكل الهندسي التنسيق المحتملة لهذه المعقدات ثماني السطوح. بعض المعقدات موصل لتيار الكهرباء -الآخرين غير موصل لتيار الكهرباء. أعدت المعقدات أيونات المعادن التالية معهما الصيغ المقترحة على ما يلي: المعقدات أيون الفلز من الليكندات (L_1 و L_2) من خلال التكثيف خليط من [الكروم (III)والمنغنيز (II)والحديد (II)والحديد (III)والكوبلت (II)والنيكل (II) والنحاس (II) والزنك (II)والكادميوم (II) والزئبق (II)] الأيونات على التوالي، في الايثانول مع التحريك أعطى الصيغ الأتية :

$[ML_1L_2\,Cl(H_2O)].2H_2O$ حيث $M: \{Hg^{2+}, Cd^{2+}, Zn^{2+}, Cu^{2+}, Ni^{2+}, Co^{2+}, Fe^{2+}, Mn^{2+}\}$

$[ML_1L_2Cl(H_2O)]Cl.xH_2O.$ حيث $M: \{Cr^{3+}, Fe^{3+}\}$ $x: \{2, 3\}$

الكلمات المفتاحية: كوينولونز (سبروفلكساسين)-2-فينايل-2-(ح-ميثوكسي انلين) اسيتونتريل ايونات العناصر الانتقالية