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Synthesis and characterization of some biological active transition metal complexes of Schiff base derived from cefixime with mixed ligand 8-hydroxy quinoline

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Abstract:

The aim of the work is synthesis and characterization of bidentate ligand [dipotassium sodium7-((E)-2-(2-((Z)-1-carboxylatoethylideneamino)thiazol-4-yl)-2 (carboxylatemethoxyimino) acet amido)-8-oxo-3-vinyl-5- thia-1-azabicyclo[4.2.0] oct-2- ene-2- carboxylate] [Nak_2L], from the reaction of cefixime with sodium pyruvet to produce the ligand [Nak_2L], the reaction was carried out in methanol as a solvent under reflux. The prepared ligand [Nak_2L] which was characterized by FT-IR, UV-Vis spectroscopy, ^1H, ^{13}C-NMR spectra, Mass spectra, (C.H.N) and melting point. The mixed ligand complexes were prepared from ligand [Nak_2L] was used as a primary ligand while 8-hydroxy quinoline [Q] was used as a secondary ligand with metal ion M(Π).Where M(Π) = (Mn ,Co ,Ni ,Cu ,Zn and Cd) at reflux ,using methanol as a solvent, KOH as a base. Complexes of the composition [$(M)_2(Q)_2(KL)(H_2O)_4$] with (2:2:1) molar ratio were prepared. All the complexes were characterized by spectroscopic methods (FT-IR, UV-Vis spectroscopy) along with elemental analysis (A.A), chloride content and melting point measurements were carried out, together with conductivity and magnetic susceptibility. These measurements showed octahedral geometry around (Mn^{II}, Co^{II}, Ni^{II}, Cu^{II}, Zn^{II} and Cd^{II}) ions. The biological activity of the ligands [NaK_2L], [Q] and complexes [$(M)_2(Q)_2(KL)(H_2O)_4$] were studied by using inhibition method.

Key words: Cefixime Schiff base, 8-hydroxyquinoline

Introduction:

Metal based drugs have occupied an important role in medical sciences some of these metal base drugs which are currently in use contain Al, As, Ca, Ge, Au, Fe, Hg, Sb, and Mg metal ions [1]. Recently several metal complexes of cefixime have been prepared. Azmi .et.al [2] .reported the complexation reaction between cefixime and palladium ion in the presence of acidic buffer solution (pH3) in ethanol- distilled water medium at room temperature. Also Juan R.Anacona and Jesus Estacio published their work on synthesis and antibacterial activity of cefixime metal complexes ,octahedral geometry were concluded from their work of cefixime metal complexes [3]. M. Saeed Arayne et.al [4], studied the antibacterial of metal complexes of cefixime, with copper zinc and cadmium have been carried out by observing the minimum inhibitory concentration (MIC) and by measuring the zone of inhibition of the complexes and compared with the parent drug. Most of these drugs have improved pharmacological properties in the form of their metal complexes. These facts motivate researcher to explore the coordination behavior of a newly synthesized drug. Although literature survey revealed that little work involving metal-based β -lactam antibiotics containing Schiff base has been reported so far. Though J .R . Anacona .et. al [5] reported the synthesis ,spectroscopic ,and magnetic studies of mono – and polynuclear Schiff base metal complexes containing salicylidene – cefotaxime ligand .The electronic spectra of the complexes and their magnetic moments suggest tetrahedral geometry for isolated complexes.

Also the second ligand 8-hydroxyquinoline (8-HQ) is a potent lipophilic metal chelator which 8-hydroxyquinoline copper chelate, CuQ 5 ,is used extensively as a fungicide in many countries [6] .Besides its antibacterial activity is long known[7]. Nowadays mixed ligand complexes are used; they are suitable for mimicking the role of metal ions, in drug designing. New mixed ligand complexes were continuously studied toget biologically active compounds. J. R. Anacona .et.al [5], studied the Metal complexes of a Schiff base ligand derived from cefotaxime and salicylaldehyde. The aim of the present work deals with the synthesis of a new cefixime Schiff base ligand [NaK_2L], then studying the complexation of some transition metal ions using mixed ligand cefixime Schiff base as a primary ligand with 8-hydroxy quinoline as a secondary ligand. different spectroscopic techniques and also elemental microanalyses were done. Antibacterial properties of ligands cefixime Schiff base [NaK_2L], 8-Hydroxyquinoline [HQ], and their complexes towards gram positive and gram negative bacteria were studied.

Materials and Methods:

All common laboratory chemicals and reagents and their suppliers have been used without further purification. Purity varied from 98% to 99.9%.

Physical measurements

The following measurements were used to characterize the ligand and its complexes. An electro thermal apparatus Stuart melting point was used to measure the melting points. FT-IR spectra were recorded by using Shimadzu, (FT-IR)-8300, Infrared Spectrophotometer in the range (4000–400) cm^{-1}. Spectra were recorded as potassium bromide discs. The electronic spectra of the compounds were obtained using Shimadzu UV-160A- Visible Recording Spectrophotometer, in the range (1100 -200 nm) using quartz cell of (1.0) cm length. The samples with concentration (10^{-3}) mole L^{-1} in DMSO at 25°C were measured.

Electrical conductivity measurements of the complexes were recorded at (25 °C) for (10^{-3}) M solutions of the samples in DMSO as a solvent using Philips pw-Digital Meter Condictivity. Elemental microanalyses were recorded by microanalysis (C.H.N) analyzer, Euro (Vector EA 3000A). Spectra for the ligand [Nak_2L] were recorded in DMSO-d^6 using Brucker, model: Ultra Shield 300 MHz, origin: Switzerland and are reported in ppm (s). The chloride contents for complexes were determined by potentiometric titration method on (686-Titro Processor-665.Dosimat Metrohm Swiss). The metal contents of the complexes were determined by atomic absorption (A.A) technique, using a Shimadzu (A.A 680 GBC 933 plus) atomic absorption spectrophotometer. A known amount of each metal complex was digested with 15ml of concentrated HNO_3 and diluted to a volume of 100ml with deionized water. Then, the metal content in the complexes were determined using atomic absorption spectroscopy [8]. The magnetic susceptibility of the complexes was obtained by using (Balance Johnson Mattey).The mass spectrum for the ligand [Nak_2L] was obtained by Electron-Impact (EI) on Shimadzu GCMSQPA 1000 spectrometer.

Synthesis

1. Synthesis of the ligand [Nak_2L]

A solution of cefixime (0.5g, 1mmole) in methanol (10) ml was added drop wise, under stirring to a solution of Sodium pyruvate (0.1g, 1mmole) dissolved in methanol (10 ml). A solution of (0.1M) KOH was added to adjust the pH at (7-8) and the reaction mixture was refluxed for (3 hrs) .The resulting product was filtered , washed with methanol. Recrystallization from methanol gave an orange color of the Schiff base, yield (82%), m.p (102-105) °C. The synthesis route of the ligand is shown in scheme (1).The microanalysis results for the ligand and some of its physical properties is given in table (1).

2. Synthesis of the mixed-ligand [NaK_2L] and 8-hydroxy quinoline complexes with some metal ions [$(M)_2(Q)_2(KL)(H_2O)_4$] Synthesis of [$(Co)_2(Q)_2(L)(H_2O)_4$] complex.

The metal solution of $CoCl_2.6H_2O$ (0.09g, 0.37 mmole) in (10) ml methanol was stirred for (10) minutes. The ligand solution (0.1 g, 0.18m mole in (10) ml methanol after adjusted to pH=8 using few drops of KOH solution was added to the metal solution. Finally a solution of 8-hydroxy quinoline (0.05, 0.34 mmole) in (10) ml methanol was also added to the above metal solution. The resulting mixture was heated under reflux for (2) hrs. Then the mixture was filtered and the precipitate was washed with an excess of methanol and dried at room temperature during (24) hrs. A grey solid was obtained. Weight (0.135g), yield (86.92%), m.p (>300°C) dec.

A similar method to that mentioned in preparation of (Co) complexes was applied for the preparation of (Mn^{II}), (Ni^{II}), (Cu^{II}), (Zn^{II}) and (Cd^{II}) complexes. The physical properties and elemental mixed ligand [$(M)_2(Q)_2(KL)(H_2O)_4$] analysis of complexes is given in table (2)

Results and Discussion:

The synthesized ligand dipotassium sodium {7-((E)-2-(2-((Z)-1-carboxylatoethylid eneamino)thiazol-4-yl)-2-(carboxylatomethoxyimino) acetamido)-8-oxo-3-vinyl-5-thia-1-azabi-cyclo[4.2.0]oct-2-ene-2-carboxylate} [Nak_2L] has been characterized by FT-IR, ^1H,^{13}C-NMR, Mass spectra, (C.H.N), UV-Vis spectroscopic methods.

1. IR spectra and NMR

The (FT.IR) spectrum of ligand [NaK_2L] shows bands at (1758cm^{-1}) and (1612cm^{-1})were signed to stretching vibration motions to υ(C=O) lactam and υ(C=O) amide of the Schiff base [NaK_2L] [9]. The bands at (1531 cm^{-1}) and (1479 cm^{-1}) were attributed to υ(C=N) out and υ(C=N) int stretches vibrations. While the (I.R) spectrum of the Schiff base [NaK_2L] shows absorption band at (1593 cm^{-1}) which can be assigned to υ(C=N) imine. The (I.R) spectrum of the Schiff base [NaK_2L] asymmetric and symmetric stretching vibrations of carboxylate group were observed at (1479 cm^{-1}) and (1257cm^{-1}) respectively[10] . Medium absorption band at (2940, 2835 cm^{-1}) which can be assigned to region corresponds to aliphatic (C–H), while aromatic (C–H) stretch appear in the (3084 cm^{-1}) region. The spectrum of the 8-hydroxy quinoline displays a band at (3182 $cm^{-1)}$ is due to υ(OH) stretching vibration [11]. The band at (1579 cm^{-1}) was assigned to the υ(C=N) stretching vibration[12].While FT-IR spectral data for the mixed ligand complexes of $[(Mn)_2(Q)_2(KL)(H_2O)_4]$ (1) ,$[(Co)_2(Q)_2(KL)(H_2O)_4]$(2) ,$[(Ni)_2(Q)_2(KL)(H_2O)_4]$(3), $[(Cu)_2(Q)_2(KL)(H_2O)_4]$(4) ,$[(Zn)_2(Q)_2(KL)(H_2O)_4]$(5) and $[(Cd)_2(Q)_2(KL)(H_2O)_4]$ (6) complexes, are summarised in Table (3). The band at (1593cm^{-1}) is due to ν(C=N) (imine) stretching vibration for the Schiff base ligand $[NaK_2L]$, but in the complexes the bands were at (1576), (1581), (1577), (1573), (1579) and (1574) cm^{-1} for the complexes (1),(2),(3),(4) ,(5) and (6), respectively, this shift to lower frequencies may be due to delocalization of metal electron density into the ligand π- system [13-14]. HOMO→LUMO (where HOMO: highest occupied molecular orbital, LUMO: lowest unoccupied molecular orbital). The shift in ν(C=N) confirms the coordination of ligand through Nitrogen atoms to the metal of ions[15].These bands were assigned to the ν(C=N) stretching of reduced bond order. The band at (1758cm^{-1}) is due to ν(C=O) (lactam) stretching vibration for the Schiff base ligand $[NaK_2L]$, but in the complexes was at (1742), (1741), (1743), (1743), (1747) and (1746) cm^{-1} for the complexes (1), (2), (3), (4), (5) and (6), showing that the coordination is through the Oxygen atom of ß-Lactam group. The band for ν(C=O) amid in the ligand $[NaK_2L]$ was at (1612cm^{-1}) which remains relatively constant in all complexes which indicates no bonding through the (C=O) amid group. The band at (1579cm^{-1}) is due to ν(C=N) stretching vibration for the mixed-ligand [8-HQ], but in the complexes was at(1497), (1499), (1498), (1498), (1499) and (1498) cm^{-1} for the complexes (1), (2), (3), (4), (5) and (6) respectively[16], showing that the coordination of the metal ion is through the Nitrogen atom of ligand [8-HQ]. The bands at (1479 cm^{-1}) and (1257 cm^{-1}) were assigned to asymmetric and symmetric stretching vibration (COO^-) for the ligand $[NaK_2L]$ respectively. On complexation these bands have been shifted to lower and higher frequencies[(1464 cm^{-1}),(1465 cm^{-1}), (1465 cm^{-1}), (1463 cm^{-1}), (1465 cm^{-1}) and,(1464 cm^{-1}) for υ_{asy}(COO)], while [(1273 cm^{-1}),(1274 cm^{-1}), (1278 cm^{-1}), (1278 cm^{-1}), (1271 cm^{-1}) and, (1278 cm^{-1}) for υ_{sy}(COO)], for the complexes (1),(2),(3),(4) ,(5) and (6) respectively,, showing that the bonding of metal ion was through the oxygen atom of carboxylate ion .Where $\Delta\upsilon = [\upsilon_{asym}(COO^-) - \upsilon_{sym}(COO^-)]$ are (191), (191), (187), (185), (194) and(196),these high values are indicating mono dentate coordination [17]. The appearance of new and medium intensity ν($O\text{-}H_2$) stretching vibration bands at (3343),(3368),(3361), (3379), (3338) and (3338) cm^{-1} for the complexes (1),(2),(3),(4) ,(5) and (6) were observed. While the (I.R) spectrum of the mixed -ligand [8-HQ] shows absorption band at (3182 cm^{-1}) which can be assigned to corresponds to υ(O-H) qu. This band disappeared upon complexation. The bands at (498,418), (485,416), (466,451), (489,412), (486,447)and (492,482)cm^{-1} were assigned to ν (M-O) stretching vibrations for the complexes (1), (2), (3), (4), (5) and (6) respectively, indicating that carboxylic oxygen and oxygen of ß-Lactam group of the ligand are involved in the complexation with metal ions. Other low intensity bands observed in the far IR region in (646,536), (650,528), (553,501), (630,582), (598,505) and

(602,573) cm^{-1} were assigned to ν (M-N) stretching vibrations for the complexes (1),(2),(3),(4) ,(5) and (6) respectively[18], indicating that the nitrogen of Schiff base ligand and nitrogen of ligand [8-HQ] , are involved in coordination with metal ions. ^{1}H-NMR spectrum of ligand [NaK_2L], showed the following characteristic chemical shift (DMSO as a solvent): A singlet signal at δ 8.1 ppm attributed to the one proton of C_{16} for (C-S) thiazole group . A singlet signal at δ 8.0 ppm attributed to the one proton of (N-H) amide group. The chemical shift at δ 6.3 due to C_6 ppm for proton of ethylene group. The chemical shift at δ 5.1-5.0 ppm due to C_5, C_7 for C-H propiolactam. A signal at δ 4.7 ppm attributed to the two proton of C_{13} for CH_2-O group. A signal at δ 3.3-3.1ppm attributed to the two proton of C_2 for S-CH_2 group. The chemical shift at δ 2.4 ppm due to DMSO solvent, and finally, a three proton of methyl group (C-H) appeared at δ 2.1 ppm [19]. The ^{13}C-NMR spectrum of a ligand [NaK_2L] reports that the carbon atom (C_{14}) of carbonyl group resonated at δ (178.4) ppm while the carbon atoms (C_8 and C_{20}) of carbonyl group resonated at δ (168.7) ppm. The carbon atom (C_{17}) of imine group resonated at δ(169.1) ppm . The carbon atoms (C_6 and C_{11}) of carbonyl group resonated at δ (164.1) ppm. The carbon atoms (C_{18} and C_{12}) of imine group resonated at δ (164.1) ppm. The carbon atom (C_{15}) of C=CH group resonated at δ(152.3) ppm while the carbon atoms (C_3 and C_9) of carbonyl group resonated at δ (128.1) ppm . The carbon atoms (C_{10}, C_{13} and C_2) of CH_2 group resonated at δ(118.1, 78.4 and 26.9) ppm respectively. The chemical shift at δ (40.0) ppm to DMSO solvent. Finally the carbon atom (C_{19}) of CH_3 group resonated at δ (18.8) ppm[20]. The FTIR, 1H-NMR and 13C-NMR spectra were confirmed the chemical structure of a new ligand [NaK_2L], table(3),(4)and(5).

2. Mass spectrum

The mass spectrum of the ligand [NaK_2L] was also consistent with the proposed structure formula. The mass spectrum of the ligand shows parent ion peak at [M/Z] =620 which correspond to[M^+], Figure (2).

3. Electronic spectra, magnetic moments and conductivity measurements

The electronic spectrum of ligand (NaK_2L), Figure (3) exhibit high intense absorption peaks at (231 nm) (49290 cm^{-1}) (ε_{max}=2126 molar^{-1}.cm^{-1}) and (295 nm) (33898 cm^{-1}) (ε_{max}=1554 molar^{-1}.cm^{-1}) which assigned to ($\pi\rightarrow\pi^*$) and ($n\rightarrow\pi^*$) transition respectively, the data recorded in Table (6), The electronic spectrum of (8-hydroxy quinoline), figure (4) exhibits a high intense absorption peak at (301 nm) (33222 cm^{-1}) (ε_{max}=2295molar^{-1}.cm^{-1}), which assigned to ($\pi\rightarrow\pi^*$) transition [21]. the data recorded in table (7)

Most of transition metal complexes are colored and their colors are different from the transition metal salts and ligands, then this is an important indication of coordination, therefore the colored complexes showed different characteristic absorption bands in their position and intensity [22]. The electronic spectrum of [$(Ni)_2 (A)_2 (KL) (H_2O)_4$], Fig.(5) showed two intense peaks in the range (273 nm),(36630 cm^{-1}), ($\in_{max}$=1975 molar^{-1}. cm^{-1}) and(301nm),(33222 cm^{-1}), ($\in_{max}$ =1885 molar^{-1}. cm^{-1}) are assigned to the ligand field ·And another intense peak in the range (394nm), (25380 cm^{-1}), ($\in_{max}$=921 molar^{-1}. cm^{-1}) due to charge transfer transition. And the peak at visible region at (892 nm), (11210 cm^{-1}), ($\in_{max}$ = 12 molar^{-1}.cm^{-1}) these speaks are assigned to ($^3A_2g\rightarrow{}^3T_1g_{(p)}$) and ($^3A_2g\rightarrow{}^3T_1g_{(F)}$) (d-

d) transitions confirming a trans octahedral structure around (Ni^{+2}) ion complex. Where a comparison between the data recorded of the mixed ligands and (Ni^{II}) metal ion is given table (8)[23].

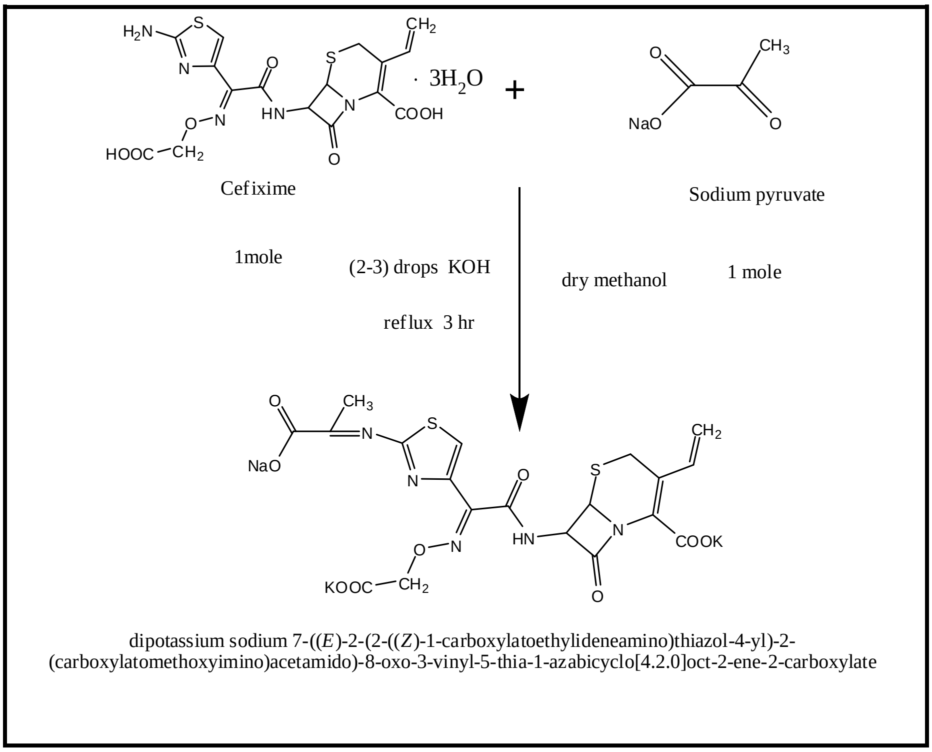
The molar conductivities indicate that all metal complexes are non-electrolytes Table (2). These data together with the magnetic moment indicate octahedral geometry around the metal atoms studied, table (2), scheme (2).

4. Biological activity studies

The synthesized ligand and its complexes were screened for their antibacterial activity [24] against Staphylococcus aureu., Bacillus subtilis, Escherichia coli and Psedomonas aeruginosa activity at (10^{-3}) mole/liter concentration. The zones of inhibitions of the antimicrobial activity have been presented in Table (9). The results of antibacterial activity of ligands and its complexes show good activity against all the bacteria used, except $[(Ni)_2(Q)_2(KL)(H2O)_4]$ with E-coli, [HQ] ligand with Pseudomonas and $[NaK_2L]$ ligand with Staphylococcus.

Conclusion:

In this paper we have explored the synthesis and coordination chemistry of some mixed ligand complexes. The mode of bonding and overall structure of the complexes were determined through physico-chemical and spectroscopic methods. The results show that the solid complexes with a ratio of M: Q: L as (2:2:1).



Scheme (1) Synthesis route of the ligand $[NaK_2L]$

Table (1) Microanalysis results and some physical properties for the ligand $[Nak_2L]$

Compound	Empirical formula	M.wt	Yield %	M.P °C	Color	Found (Calc.) %		
						C	H	N
$[Nak_2L]$	$C_{19}H_{14}K_2N_5NaO_9S_2$	621.6	80	(102-105)	Pale yellow	36.71 (35.28)	2.27 (2.15)	11.27 (10.54)

M.P= melting point; Calc= Calculated

Table (2) some physical properties of the prepared mixed- ligand complexes $[(M)_2(Q)_2(KL)(H_2O)_4]$ together with their magnetic moment and molar conductivity measurement

No.	Empirical formula	Color	Dec. C^O	M	Cl %	Yield %	magnetic moment	Λ_m $S.cm^2$ $molar^{-1}$
1	$[(Mn)_2(Q)_2(KL)(H_2O)_4]$	Dark yellow	> 300	11.25 (12.06)	Nil	90.9	5.71	8.55
2	$[(Co)_2(Q)_2(KL)(H_2O)_4]$	Grey	> 300	12.98 (12.82)	Nil	86.9	4.43	8.71
3	$[(Ni)_2(Q)_2(KL)(H_2O)_4]$	Pale green	> 300	12.33 (12.78)	Nil	77.3	2.82	11.3
4	$[(Cu_2(Q)_2(KL)(H_2O)_4]$	Dark green	> 300	14.05	Nil	70.1	1.75	2.85
5	$[(Zn)_2(Q)_2(KL)(H_2O)_4]$	Pale yellow	> 300	(13.69)	Nil	69.8	---	6.31
6	$[(Cd)_2(Q)_2(KL)(H_2O)_4]$	yellow	> 300	14.61	Nil	92.7	---	5.16

Dec. =Decomposition

Table (3) FT-IR spectral data (wave number υ⁻) cm^{-1} of the mixed-ligand complexes with some metal ions

Compound	υ(OH_2) water	υ(OH)qu	υ(C=O)lactm	υ(C=N)imine	υ(C=O)amid.	υ(C=N) qu.	$υ_{as}(COO)$ $υ_s(COO)$	Δcm^{-1}	υ(M–N)	υ(M–O)
$[NaK_2L]$	– –	-	1758	1593	1612	–	1479 1257	–	–	–
8-HQ	– –	3182	– –	–	–	– 1579	– –	–	–	–
$[(Mn)_2(Q)_2(KL)(H_2O)_4]$	3343	-	1742	1576	1616	1497	1464 1273	191	646 536	498 418
$[(Co)_2(Q)_2(KL)(H_2O)_4]$	3368	-	1741	1581	1617	1499	1465 1274	191	650 528	485 416
$[(Ni)_2(Q)_2(KL)(H_2O)_4]$	3361	-	1743	1577	1604	1498	1465 1278	187	553 501	466 451
$[(Cu_2(Q)_2(KL)(H_2O)_4]$	3379	-	1743	1598	1618	1498	1463 1278	185	630 582	489 412
$[(Zn)_2(Q)_2(KL)(H_2O)_4]$	3338	-	1747	1579	1618	1499	1465 1271	194	598 505	486 447
$[(Cd)_2(Q)_2(KL)(H_2O)_4]$	3338	-	1746	1574	1615	1498	1464 1268	196	602 573	492 482

Recorded as KBr disc, υ(OH)qu. = for 8-HQ , υ(C=N)qu.= for 8-HQ

Table (4) ^1H-NMR data for ligand measured in DMSO-d^6 and chemical Shift in ppm (δ)

Compound	Functional groups	δ (ppm)
$[NaK_2L]$	C_{16} for C-S thiazole group	(8.1) (1H, s)
	N-H	(8.0) (H, s)
	C_9 for C-H ethylene	(6.3) (1H,s)
	C_5, C_7 for C-H propiolactam	(5.1, 5.0) (1H,1H, s)
	C_{13} for CH_2-O group	(4.7) (2H,s)
	C_2 for S-CH_2 group	(3.3, 3.1) (2H,m)
	DMSO solvent	(2.4)
	C_{19} for CH_3 group	(2.1) (3H, s)

s=single, m=multiple

Table (5) ^{13}C-NMR data for ligand measured in DMSO-d^6 and chemical Shift in ppm (δ)

Compound	Functional groups	δ (ppm)
$[NaK_2L]$	$C_{14}$for C=O group	178.4
	C_{17} for C=N group	169.1
	C_8, C_{20} for C=O group	168.7
	C_6, C_{11} for C=O group	164.1
	C_{18}, C_{12} for C=N group	164.1
	$C_{15}$for C=CH group	152.3
	C_3, C_9 for C=CH_2 group	128.1
	C_{10} for CH_2 group	118.1
	C_{13} for CH_2 groups	78.4
	C_5, C_7 for C-H groups	52.3, 48.5
	DMSO solvent	40.0
	C_2 for CH_2 group	26.9
	C_{19} for CH_3 group	18.8

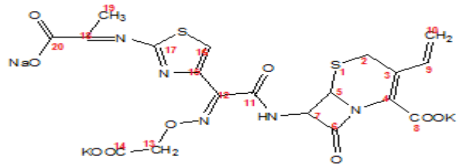


Fig.(1) structure of Schiff base $[NaK_2L]$

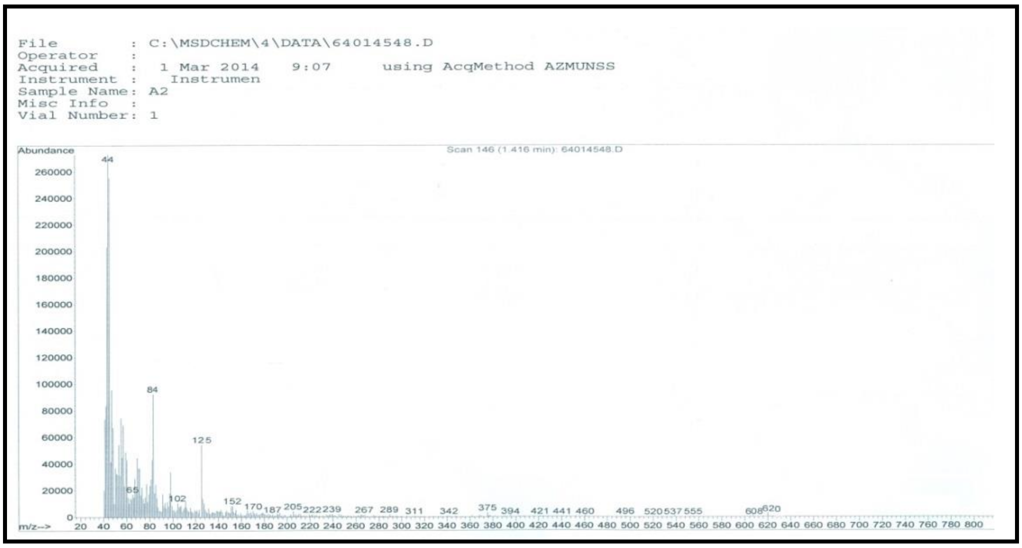


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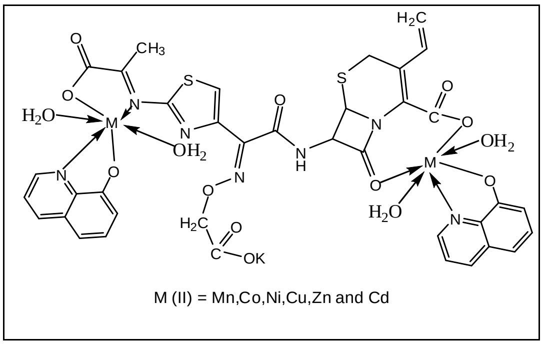
Fig.(2) EI-mass spectrum for the ligand [Nak_2L]



Scheme (2) A proposed structure of the complexes [$(M)_2(Q)_2(KL)(H_2O)_4$]

Table (6) Electronic spectral data of the ligand [NaK_2L]

Compound	λ (nm)	$\bar{\upsilon}$ cm^{-1}	ε_{max} molar^{-1}cm^{-1}	assignments
[NaK_2L]	231	49290	2126	$\pi\rightarrow\pi^*$
	295	33898	1554	$n\rightarrow\pi^*$

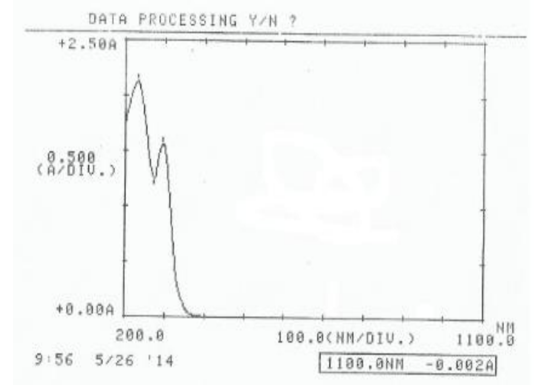


Fig.(3): The Electronic spectrum of the [NaK_2L]

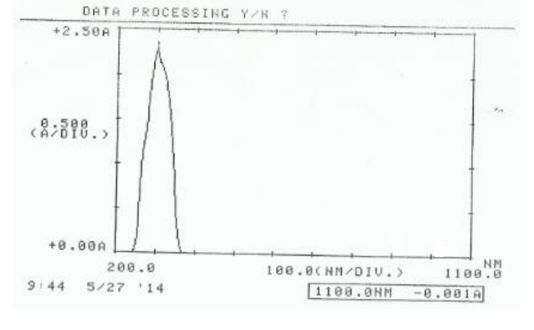


Fig. (4): The Electronic spectrum of the (HQ)

Table (7) Electronic spectral data of the [HQ]

Compound	λ (nm)	$\bar{\upsilon}$ cm^{-1}	ε_{max} molar^{-1} cm^{-1}	assignments
[HQ]	301	33222	2295	$\pi\rightarrow\pi^*$

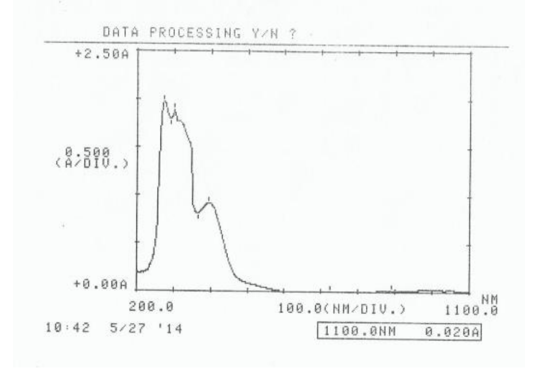


Fig. (5): The Electronic spectrum of the [$(Ni)_2(Q)_2(L)(H_2O)_4$]

Table (8) Electronic spectral data of metal complexes $[(M)_2(Q)_2(KL)(H_2O)_4]$

No.	Compound	Wave number		ε_{max}	Assignment	Suggested structure
		nm	cm^{-1}	$molar^{-1}\ cm^{-1}$		
1	$[(Mn)_2(Q)_2(KL)(H_2O)_4]$	274	36496	1989	L.F	Oh
		301	33222	1789		
		382	26178	787	C.T	
		947	10559	8	$^6A_1g_{(S)}\rightarrow{}^4T_1g_{(G)},{}^4T_2g_{(G)}$	
2	$[(Co)_2(Q)_2(KL)(H_2O)_4]$	273	36630	1931	L.F	Oh
		372	26881	677	C.T	
		403	24813	732	$^4T_1g_{(P)}\rightarrow{}^4T_2g$ $^4T_1g_{(F)}\rightarrow{}^4T_2g$	
3	$[(Ni)_2(Q)_2(KL)(H_2O)_4]$	273	36630	1975	L.F	Oh
		301	33222	1885	L.F	
		394	25380	921	C.T	
		724	13812	16	$^3A_2g\rightarrow{}^3T_1g_{(P)}$	
		892	11210	12	$^3A_2g\rightarrow{}^3T_1g_{(F)}$	
4	$[(Cu)_2(Q)_2(KL)(H_2O)_4]$	278	35971	2362	L.F	Oh
		319	31347	2103		
		406	24630	1814	C.T	
		954	10482	17	$^2B_1g\rightarrow{}^2A_1g$	
5	$[(Zn)_2(Q)_2(KL)(H_2O)_4]$	272	36764	1902	L.F L.F	Oh
		301	33222	1729		
		331	30211	523		
		397	25188	1115	C.T	
6	$[(Cd)_2(Q)_2(KL)(H_2O)_4]$	277	36101	2192	L.F	Oh
		301	33222	2148		
		343	29154	567		
		397	25188	974	C.T	

Table (9): The Biological activity of the prepared ligands (Zone of inhibition (mm))

No	Compound	*E-coli*	*Pseudomonas*	*Staphylococcus*	*Bacillus*
C	DMSO	_ _	10	_	_ 6
1	NaK_2L	10	14	_	12
2	HQ	18	_	31	25
3	$[(Mn)_2(Q)_2(KL)(H_2O)_4]$	10	13	14	10
4	$[(Co)_2(Q)_2(KL)(H_2O)_4]$	15	15	12	11
5	$[(Ni)_2(Q)_2(KL)(H_2O)_4]$	0	13	14	15
6	$[(Cu_2(Q)_2(KL)(H_2O)_4]$	16	11	17	17
7	$[(Zn)_2(Q)_2(KL)(H_2O)_4]$	12	10	15	19
8	$[(Cd)_2(Q)_2(KL)(H_2O)_4]$	17	18	16	20

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تحضيروتشخيص لبعض معقدات العناصر الانتقالية النشطة حيوياً لقواعد شيف مشتقه من السيفكسيم مع مزيج من لليكاند 8-هيدروكسي كوينولين

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قسم الكيمياء ،كليه تربيه للعلوم الصرفة ابن الهيثم ،جامعة بغداد

الخلاصـــة:

أن الهدف من العمل تخليق وتشخيص ليكاند احادي السن dipotassium sodium 7-((E)-2-(2-((Z)-1-carboxylatoethylideneamino)thiazol-4-yl)-2-(carboxylatomethoxyimino)acetamido)-8-oxo-3-vinyl-5-thia-1-azabicyclo[4.2.0]oct-2-ene-2-carboxylate.$[NaK_2L]$

من تفاعل cefixime مع sodium pyruvate لينتج الليكاند $[NaK_2L]$، أجري التفاعل باستخدام ميثانول الجاف كمذيب وبطريقة التصعيد الحراري، تم تشخيص الليكاند المحضر باستخدام اطياف الاشعة تحت الحمراء واطياف الاشعة فوق البنفسجية وطيف الرنين النووي المغناطيسي للبروتون وللكاربون13 وطيف الكتلة والتحليل الدقيق للعناصر ودرجة الانصهار.حضرت معقدات مزج الليكاند تم استخدام الليكاند $[NaK_2L]$ كليكاند اول بينما 8-hydroxy quinoline كليكاند ثاني مع M(II) .

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

بطريقة التصعيد الحراري باستخدام الميثانول كمذيب، هيدروكسيد البوتاسيوم كقاعدة، بالصيغة $[(M)_2(Q)_2(KL)(H_2O)_4]$ بنسبة مولية (2:2:1). جميع هذه المعقدات المحضرة شخصت بواسطة الطرق الطيفية (اطياف الاشعة تحت الحمراء و اطياف الاشعة فوق البنفسجية والتحليل الدقيق للعناصر ونسبة محتوى الكلور وقياسات درجة الانصهار مع قياسات التوصيلية والحساسية المغناطيسية. وتم أستنتاج الشكل الهندسي ثماني السطوح حول الايونات الفلزية (Mn^{II} ,Co^{II} ,Ni^{II} ,Cu^{II} ,Zn^{II} and Cd^{II}) . تم دراسة الفعالية البايولوجية لليكاند$[NaK_2L]$ و لمعقدات $[(M)_2(Q)_2(KL)(H_2O)_4]$ اباستخدام طريقة التثبيط.

الكلمات المفتاحيه: قواعد شيف سيفكسيم ،8- هيدروكسي كينولين