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Synthesis and Characterization of metal complexes with ligands containing a hetero (N) atom and (hydroxyl or carboxyl) group

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ABSTRACT

The research includes the synthesis and identification of two types of mixed ligands complexes of M(II) Ions using amino acid L- proline as a primary ligand and either Nicotinamide or 8-hydroxyqinoline as secondary ligand, respectively:

a. The mixed ligand complexes of composition, [M(pro)₂(na)₂].

b. The mixed ligand complexes of composition, $Na[M(pro)_2(Q)]$.

Where proline ($C_5H_9NO_2$) symbolized as pro H , Nicotinamide ($C_6H_6N_2O$) symbolized as (NA) , 8-hydroxyqinoline, ($C_9H_7NO_2$) symbolized as (8-HQ).

The ligands and the metal chlorides were brought into reaction at room temperature $(37^{\circ}c)$ in ethanol as solvent .The reaction required the following molar ratios [(1:2:2) metal:2NA:2pro⁻] and [(1:1:2) metal:Q:2pro⁻] with M⁺² ions, where M = [Mn (II), Co(II), Ni(II), Cu(II), Zn(II), Cd(II) and pd(II)].

Products were found to be solid crystalline complexes, which have been Characterized through the following techniques:

Melting points, Solubility, Molar conductivity. Determination the percentage of the metal in the complexes by (AAS). Spectroscopic Methods [FT-IR and UV-Vis], and. The proposed structure of the complexes using program, chem office 3D(2006).

Key words: Amino Acid, , 8- hydroxyqinoline, L- proline, Mixed Ligand Complexes

الملخص باللغة العربية

[M(pro)₂(na)₂] -1 معقدات مختلطة الليكاند بالصيغة:

[M(pro)₂(Q)]⁻. معقدات مختلطة الليكاند بالصيغة: ...-2

(C₉H₇NO) إذ ان البرولين (C₅H₉NO₂) بالرمز (na) و 8⁻هايدروكسي كوينولين (C₆H₆N₂O) بالرمز (na) و 8⁻هايدروكسي كوينولين (C₉H₇NO) بالرمز (E₉H₀NO₂) و (C₉H₇NO) بالرمز (B-HQ) و ذلك بمفاعلة الليكاندات مع كلوريدات العناصر باستعمال الايثانول مذيبا وفي درجة حرارة المختبروبنسب مولية $M^{+2} = [Mn^{+2}, Co^{+2}, Ni^{+2}, Cu^{+2}, i + 1]$ مع M^{+2} إذ إن (1:2:2) (metal): 2na: 2pro⁻¹] و [-2na⁺², Cd⁺², pd⁺²].

المعقدات المحضرة بلورات صلبة بعضها ملون درست من النواحي الآتية: درجات الانصهار، التوصيلية الكهربائية المولارية، الذوبانية، تقدير النسبة المئوية للأيون الفلزي في المعقدات بوساطة مطيافية الامتصاص الذري ، الدراسات الطيفية: وتضمنت أطياف(الأشعة تحت الحمراء، الأشعة فوق البنفسجية– المرئية، مع استخدام برنامج (100 Chem. Office Cs – 3D pro) في رسم أشكال المعقدات

INTRODUCTION

8-hydroxyqinoline or 8-qinoline is the name most commonly used ,while its trivial name is (oxine) ,which is conventionally used for the description of chelate compounds Oxinates ,as out of seven possible hydroxyginoles, only 8hydroxyginoline forms chelate with metal ions. (1). 8-hydroxyqinoline is wellcharacterized organic chelating ligand ,which can form covalent compounds with over 60metal ions under controlled pH-conditions ,and its preference for transition and heavy metal cations over alkali and alkaline-earth cations is well known. (2).

8 -hydroxyginoline has a wide variety of uses and its medicinal and agricultural Significances were discovered before the start of current (3,4). A series of mixed- ligand (saccharinato, amino acids, Iminodiacetic acid and dimethylglyoxime) complexes of Cu(II), Co(II), Ni(II) and Zn(II) were recently reported. (5-9) .Ganesh and Co-worker[10] were prepared and characterized of mixed ligand dioxand thorium (IV) complexes ouranium(VI) of 8-hydroxyquinoline as a primary ligand and amino acids such as L-proline (ProH) and 4hydroxy-L-proline (Hyp) as secondary ligands. These complexes have been screened for their antibacterial and cyto-toxic (IC50) characteristic properties .

In this paper we reported the synthesis ,spectroscopic and structural of two types mixed ligand complexes of M(II) ions using amino acid proline as a primary ligand and either nicotinamide or 8-hydroxyqinoline as secondary ligand, respectively.

MATERIALS AND METHODS

Reagents: L-proline ,nicotinamide and 8hydroxyqinoline were purchased from (Merck), metals chloride and solvents from (B.D.H). The reagents were applied without further purification.

b-Instruments: UV-Vis spectra were recorded on a (Shimadzu UV- 160A) Ultra Violet-Visible Spectrophotometer. FTI R- 8400S) Fourier Transform Infrared Spectrophotometer (4000-400) cm⁻¹ with samples prepared as K_Br discs. Metal contents of the complexes were determined by atomic absorption technique using a Shimadzu AA 680G atomic absorption spectrophotometer. Conductivities were measured for 10⁻³M of complexes in (DMF) at 25°C using (Philips PW- Digital Conduct meter). Magnetic measurements were recorded on a Bruker BM6 instrument at 298°K following the Faraday's method . In addition melting points were obtained using (Stuart Melting Point Apparatus). The proposed molecular structure of the complexes were drawing by using chem. proposed models of the species were built with chem. 3DX (2006).

Synthesis method:

A) Sodium-8-oxyqinoliney (8Q): dissolve [0.145 gm,1mmol]8- H ydroxyquinoline(QH)with[0.04gm1mmol)] sodium hydroxide in (10ml) ethanol was deprotonated according to the following reaction: Scheme (1)



Scheme (1): Synthesis of the sodium-8oxyqinolinate

B) Sodium prolinate (Pro⁻ Na⁺): L-proline [0.230 gm (2mmol)] was dissolved in 10 ml ethanol and added to 10 ml of ethanolic solution containing [0.08 gm (2mmol)] of the sodium hydroxide , the solution was deprotonated according to the following reaction:



C) Nicotinamide solution: Nicotinamide[(0.112 gm) 1m.mol] was dissolved in 10 ml ethanol.

D) Synthesis of complexes: An aqueous solution of the metal salt was added to the solution of the ligand in ethanol respectively using stoichiometric amounts [(1:2:2)(metal):2NA:2pro⁻] and [(1:1:2)] metal:Q:2pro⁻] molar ratio , the mixture was stirred for half an hour at room temperature, crystalline precipitates observed. The resulting precipitates were filtered off, recrystallized from ethanol and dried at room temperature.

RESULTS AND DISCUSSION

Products were found to be solid crystalline complexes, which have been characterized through the following techniques: Solubility, Molar conductivity. Determination the percentage of the metal in the complexes by (AAS). Spectroscopic Method [FT-IR and UV-Vis], Biological effects for some complexes were investigated and Program [Chem. office .CS. Chem. 3D pro 2006 was used. The Physical properties listed in Table (1). All the complexes are colored, non-hygroscopic, and appears as powders with high melting points .They are not soluble in water. All complexes dissolved in DMF and DMSO solvents. The atomic absorption measurements Table (1) for all complexes gave approximated values for theoretical values.

Table (1). Analytical data and some physical properties of the complexes

Compounds	M.wt	Color	M.p°c (de)°c	*М.С µS.cm ⁻¹	Metal% theory	Metal % Exp.
$[Mn(C_6H_6N_2O)_2(C_5H_8NO_2)_2]$	549	White	299(dec)	13.27	10.43	11.24
$[Co(C_6H_6N_2O)_2(C_5H_8NO_2)_2]$	530.19	Pale- Brown	277(dec)	15.15	11.11	12.4
$[Ni(C_6H_6N_2O)_2(C_5H_8NO_2)_2]$	530.96	Pale- blue	290(dec)	29.11	11.55	11
$[Cu(C_6H_6N_2O)_2(C_5H_8NO_2)_2]$	535.71	Pale- blue	>300 (dec)	19.18	11.84	11
$[Zn(C_6H_6N_2O)_2(C_5H_8NO_2)_2]$	537.63	White	>300(dec)	17.43	12.17	11
$[Cd(C_6H_6N_2O)_2(C_5H_8NO_2)_2]$	584.6	White	>300 (dec)	25.58	19.22	21
$[Pd(C_6H_6N_2O)_2(C_5H_8NO_2)_2]$	578.6	Red	>300 (dec)	32.67	18.38	20
$Na[Mn(C_9H_6NO)(C_5H_8NO_2)_2]$	450.25	Yellow	270 -300 (dec)	68.1	12.19	14
$Na[Co(C_9H_6NO)(C_5H_8NO_2)_2]$	454.25	Green wish - Brown	>300 (dec)	71.6	12.98	14.8
$Na[Ni(C_9H_6NO)(C_5H_8NO_2)_2]$	454.11	Green	280(dec)	83.31	12.92	11.92
Na[Cu(C ₉ H ₆ NO)(C ₅ H ₈ NO ₂) ₂]	458.77	Greenwish- Brown	>300 (dec)	77.7	13.85	14.21
$Na[Zn(C_9H_6NO)(C_5H_8NO_2)_2]$	460.69	Yellow	290(dec)	86.3	14.18	15.88
Na[Cd(C ₉ H ₆ NO)(C ₅ H ₈ NO ₂) ₂	507.40	White	245(dec)	88.2	22.15	23.66
$Na[Pd(C_9H_6NO)(C_5H_8NO_2)_2]$	501.4	Red	292(dec)	73.4	21.8	23.9

M.C = Molar Conductivity, (de) = decomposition

The molar conductance values of the complexes in DMF at 10^{-3} M concentration are found to be (13.27. -32.67) μ S.cm⁻¹, for complexes of composition [M^{II}(Pro)₂(NA)₂] indicating their non-electrolytic nature while the complexes of composition Na[M^{II}(Pro)₂(Q)](68.1 -- 88.2) μ S.cm⁻¹are found to be correspond to 1:1 electrolytes.(11)

The analytical data shown in Table (1) confirmed the following molar ratios $[(1:2:2)(metal):2NA:2pro^{-}]$ and $[(1:1:2)(metal):Q:2pro^{-}]$ with M⁺² ions. The infrared spectra of various mixed ligand complexes synthesized are compiled in

tables (2& 3) figures (1-5). The infrared spectra of these complexes in comparison with free ligands show characteristic band positions, shifts and intensities, which can be correlated to monodentate nicotinamide [2,13] binding and bidentate (Proline acid and 8-hydroxyqinoline) chelation. The deprotonated ligand (Proline acid) to (Prolinate ion) (Pro⁻) by using (Na OH) coordinated to metal ions as bidentate ligand through the oxygen atom of the carboxylate group (-COO⁻) and the nitrogen atom of the imine group(NH). The nicotinamide coordinated as a monodentate through the nitrogen of the pyridine group.

Compound	NH _{2asy} str	NH str	CH (py) str	C=O str(amid)	NH ₂ (am)	C- Ncyc	(C-C) str (py)	C=N Str (am)	O=CN Bend (am)	ບ (-CO asym	O⁻) sym	M-N	М-О
Nicotinamide	3368vs	3161s	3060sh	1679vs 1697sh	1618vs	-	1423vs 1123m	1395vs	736vs	-	_	_	I
Proline	-	3056	-	Ι	I	1473 946vs	_	_	I	1600-1624	1450s -1377	_	I
Mn(NA) ₂ (pro) ₂	3394s	3309s	3190vs	1666vs	1624 vs	1577w 941w	1436m	1396s	759m	1624s 1600m	1446m 1396s	590br- m	459w
Co(NA) ₂ (pro) ₂	3402s	3194m	2924m	1670vs	1614vs	1580w 941w	1458s 1199m	1320m	752vs	1665 1610	1446s 1396s	582sh- m	418m
Ni(NA) ₂ (pro) ₂	3371vs	3174	2924m	1677m	1616vs	1456m	1458s 1199w	1326m	756m	1705vs- 1654w	1388 1476	520m	439m
Cu(NA) ₂ (pro) ₂	3402vs	3309w	2360 vs	1668m	1623vs	1485w 931m	1406s 1157m	1383m	775m	1701s 1624s	1477 1377vs	543s	430s
Zn(NA) ₂ (pro) ₂	3332br	2981m	2921S	1670m	1614vs	941s	1396s	1330m	797s	1666 -1624s	1396s m1330	568m	450m
Cd(NA) ₂ (pro) ₂	3394s	3263m	2981m	1630m	1622vs	1577m 941m	1430w	1379m	759m	1705vs 1658m	1446S 1396s	590m	459w
Pd(NA) ₂ (pro) ₂	3394vs	3159s	2924m	1636m	1618vs	1567m 937w	1488s 1166w	1354s	771s	1623-1509	1457vs 1377vs	540s	448m

Table (2) : FT-IR spectral data of mixed ligand complexes of composition, [M(pro)₂ (NA)₂]

Sym: symmetric, asy: asymmetric, am: amide, py: pyridine, o.p: out of plane,str: stretching,v.s: very strong, s: strong, m: medium, w: week, sh: shoulder.



Figure (1): FT-IR spectrum of (C₉H₇NO₂)



Figure. (2) FT-IR spectrum of (C₅H₉NO₂)



Figure (3) FTIR spectrum of (C₆H₆N₂O)



Figure (4): FTIR spectrum of [Zn(C₆H₆N₂O)₂(C₅H₈NO₂)₂]



Figure (5): UV-vis absorption spectrum of the (C₅H₉NO₂)

Compounds	NH _{sym} str	CH _(py) str	(CH) _{cyci} 1	υ (C=N)	υ (C=C)	υ(C- Ο)	υ ∂(C- Ο)	v(-C asym	OO ⁻) sym	M-N	M-0
8-HQ	-3240 3047br			1577	1508						
Na[Mn(pro) ₂ (Q)]	3174s	3136s 2854w	2974m	1620w	1573v s	1283m	457m	1465s	1323s	640 m	450 m
Na[Co(pro) ₂ (Q)]	3194br -s	3136 m 2854w	2924w	1590s	1570s	1269m	505m	1465 v s	1377vs	648 m	450w
Na[Ni(pro) ₂ (Q)]	3352	3278 br-s	2927s	1620v s	1520w	1284m	497m	1427vs	1377s	645 m	420w
Na[Cu(pro) ₂ (Q)]	3356s	3062 m	2924w	1581v s	1504v s	1289v s	468s	1469vs	1377vs	632 m	420 m
Na[Zn(pro) ₂ (Q)]	3356s	3052 m	2924sh- m	1581v s	1508m	1284m	505s	1496vs -1462 vs	1373vs - 1315vs	645 m	420w
Na[Cd(pro) ₂ (Q)]	3194br -s	3190 m	2924sh-	1573v s	1500s	1284m	505s	1496s- 1419m	1384vs - 1315vs	640 m	436w
Na[Pd(pro) ₂ (Q)]	3194br -s	3055 m	2927w	1620m	1577v s	1273m	597v s	1496s- 1465vs	1384vs - 1323vs	630 m	436w

Table (3) : FT-IR spectral data of mixed ligand complexes of composition Na [M(pro)₂(Q)]

Sym: symmetric, asy: asymmetric, str: stretching, vs: very strong, s: strong , m: medium, w:week,

The deprotonated ligand (8-hydroxyqinoline) to oxyqinolinate ion (Q^2) by using (NaOH) coordinated to metal ions as bidentate ligand through the oxygen atom of the hydroxyl group (–HO), and the nitrogen atom of pyridine group (15-17) as shown in scheme (2)



Scheme (2) The ligands coordinated

The infra red spectrum of free ligand (proline)exhibited a strong band around 3056 cm⁻¹ corresponds to the stretching vibration of v(N-H) + v(O-H), while another strong absorption band at (1600-1624) cm⁻¹ is appeared which could explained as $\upsilon(COO^{-})$ asym where the $\upsilon(COO^{-})$ symmetric stretching vibration of proline or sodium proline was assigned band at (1450-1377) cm⁻¹ (18). The infra red spectrum of free ligand (Nicotinamide) exhibited a strong band around (3368) cm⁻¹ that corresponds to the stretching vibration of v (N-H₂) .An important feature of infra-red spectra of metal complexes with 8-HQ is the absence of the band at (3240 - 3047) cm⁻¹ due to the O-H stretching vibration of the OH group of HQ (18) Figure (1). This observation leads to the conclusion that the complex formation takes place by deprotonation of the hydroxyl group of HQ moiety .Charles et al. (19) reported that for several metal complexes with HO, the υ (C-O) band is observed at 1120 cm⁻¹. The position of this band varies depending on metal complex under study. A strong υ (C-O) band observed at ~ 1104 cm⁻ indicates the presence of oxine moiety in the complexes coordinated through its nitrogen and oxygen atoms as uni negative bidentate ligand (17,18). The v (C=N)mode observed at 1577cm⁻¹in the spectra of free HO ligand is found to be shifted to lower wave number i.e. ~1500-1497 cm⁻¹ in the spectra of complexes. A negative shift in this vibration mode on complexation indicates the coordination through the tertiary nitrogen donor of HQ. Some new bands of weak intensity observed in the regions around (520-648) cm⁻¹ and (418-552) cm⁻¹ may be ascribed to M-N and M-O vibrations, respectively[17,18]. It may be noted that, these vibration bands are absent in the spectra of the ligands.

The electronic spectra:

The electronic spectra of all compounds (Ligands and complexes) are listed in Table (4) Figures. (6-8) together with the proposed assignments and suggested geometries (19, 20)

The (UV–Vis) spectra of the ligands and their complexes were studied in order to elucidate the spatial arrangement of the ligands around the metal ion.

On the basis of the above analysis (AAS, molar conductance, FT–IR, UV–Vis) spectra. The existence of hexa coordinated $M(\text{pro})_2(\text{NA})_2$. and Na [$M(\text{pro})_2(\text{Q})$].



Figure (6) UV-vis absorption spectrum of the $(C_6H_6N_2O)$



Figure (7) UV-vis absorption spectrum of the (C₉H₇NO)



Figure (8) UV-vis absorption spectrum of the [Co(C₆H₆N₂O)₂(C₅H₈NO₂)₂]

Compounds	Bacillus	Klebsiella S.P.P	Staph S.P.P
Control(DMF)	10.1	10	9
$[Ni(pro)_2(na)_2]$	19.1	15.9	11.1
$[Co(pro)_2(na)_2]$	25.4	15.9	19.1
Na[Cu(pro) ₂ (oxine)]	20.5	14.9	25.6
Na[Pd(pro) ₂ (oxine)]	20.2	18.1	20.2

 Table (4). Electronic data for the ligands and there complexes

Proposed molecular structure

Studying complexes on bases of the above analysis , the existence of Hexa coordinated [M $(C_6H_6N_2O)_2(C_5H_8NO_2)_2$] and Na[M $(C_9H_6NO)(C_5H_8NO_2)_2$], where M = [Mn (II), Co(II), Ni(II),Cu(II), Zn(II), Cd(II) and pd(II)]. A proposed models of the species were built with chem. office program [21]: Figure (9)





Figure (9) shows the antimicrobial activity of complexes (appear the inhibition zones against some pathogenic bacteria) (a) Bacillus (b) Klebsiella S.P.P (c) Staphylococcus S.P

 Table (5) Showed the inhibition circle diameter in millimeter for the bacteria after 24 hour incubation paid and 37°C for some complexes

Compounds	λ(nm)	<i>Ū</i> (cm⁻¹)	ABS	ε _(max) L.mol ⁻ . cm ⁻¹	Assignment
Proline	332	30120	0.150	150	$n \rightarrow \pi^*$
Nicotineamide	276	36238	0.620	629	$\pi \rightarrow \pi^*$
8-Hydroxyqinoline	280	35714	1.880	1880	$n \rightarrow \pi^*$
	301	33222	2.407	2407	$\pi \rightarrow \pi^*$
$[Mn(C_6H_6N_2O)_2(C_5H_8NO_2)_2]$	279	35842	1.377	1377	Ligand field
$[Co(C_6H_6N_2O)_2(C_5H_8NO_2)_2]$	276	36231	1.200	1200	Ligand field
	608	16447	0.262	262	${}^{4}T_{1g} \rightarrow {}^{4}A_{2g}$
	672	14880	0.391	391	${}^{4}T_{1g} \rightarrow {}^{4}T_{2g}(P)$
[Ni(C ₆ H ₆ N ₂ O) ₂ (C ₅ H ₈ NO ₂) ₂]	280	35714	1.624	1624	Ligand field
	390	25641	0.616	616	${}^{3}A_{2g} \rightarrow {}^{3}T_{2g}(P)$
	623	16051	0.437	437	${}^{3}A_{2g} \rightarrow {}^{3}T_{1g}$
[Cu(C ₆ H ₆ N ₂ O) ₂ (C ₅ H ₈ NO ₂) ₂]	299	33444	1.885	1885	Ligand field
	439	22779	1.48	148	${}^{2}B_{1g} \rightarrow {}^{2}A_{2g}$
	815	12269	0.065	65	${}^{2}B_{1g} \rightarrow {}^{2}B_{2g}$
$[Zn(C_6H_6N_2O)_2(C_5H_8NO_2)_2]$	273	36630	0.959	959	Ligand field
	344	29069	0.210	210	C.T
$[Cd(C_6H_6N_2O)_2(C_5H_8NO_2)_2]$	277	36101	1.240	1240	Ligand field
$[Pd(C_6H_6N_2O)_2(C_5H_8NO_2)_2]$	300	33333	2.332	2332	Ligand field
	412	24271	1.331	1331	C.T
Na[Mn(C ₉ H ₆ NO)(C ₅ H ₈ NO ₂) ₂]	279	35842	1.378	1378	Ligand field
	345	28985	1.872	1872	${}^{6}A_{1g} \rightarrow {}^{4}T_{1g}(D)$
	411	24330	1.766	1766	${}^{6}A_{1g} \rightarrow {}^{4}E_{1g}{}^{4}A_{1g}(G)$
	850	11764	0.032	0032	${}^{6}A_{1g} \rightarrow {}^{4}T_{2g}(G)$
$Na[Co(C_9H_6NO)(C_5H_8NO_2)_2]$	301	33222	1.91	1931	Ligand field
	420	23809	1.84	1840	$T_{1g}(F) \rightarrow {}^{4}T_{2g}(P)$
Na[Ni(C ₉ H ₆ NO)(C ₅ H ₈ NO ₂) ₂]	276	36231	1.192	1192	Ligand field
	328	30487	1.645	1645	${}^{3}A_{2g} \rightarrow {}^{3}T_{2g}(F)$

	388 525	25773 19047	1.100 2.20	1100 220	${}^{3}A_{2g} \rightarrow {}^{3}T_{1g}(F)$ ${}^{3}A_{2g} \rightarrow {}^{3}T_{1g}(P)$
Na[Cu(C ₉ H ₆ NO)(C ₅ H ₈ NO ₂) ₂]	281	35587	1.470	31	Ligand field
	412	24271	1.426	1426	${}^{2}B_{1g} \rightarrow {}^{2}A_{2g}$
	688	14534	0.031	1470	${}^{2}B_{1g} \rightarrow {}^{2}B_{2g}$
$Na[Zn(C_9H_6NO)(C_5H_8NO_2)_2]$	276	36231	1.188	1188	Ligand field
	332	31055	2.295	2295	Ligand field
	412	24271	1.786	1786	C.T
$Na[Cd(C_9H_6NO)(C_5H_8NO_2)_2]$	301	33222	2.286	2286	Ligand field
	390	25641	1.105	1105	C.T
Na[Pd(C ₉ H ₆ NO)(C ₅ H ₈ NO ₂) ₂]	288	34722	1 .629	1629	Ligand field
	440	22727	1.993	1993	C.T

Biological activity

The antibacterial activity of the synthesized some complexes was determined in vitro using paper disc method (agar plate diffusion method) against three pathogenic microorganism viz., Staphylococcus aureus (Gram +ve), Klebsiella S.P.P and Bacillus cereus (Gram +ve).

The solvent used was dimethyl sulfoxide (DMSO)and sample from 1 to 200 μ g/ml were used. The plates were incubated for 24 hours at 37 C⁰ (22,23)[•]. The zone inhibition of bacterial growth were measured in mm depending upon the diameter as shown in Table (5) Figure (9).

Complexes have been observed that the metal complexes have a high activity than ligand

against same organisms under the identical experimental condition.

It is evident from the above data that the antibacterial activity significantly increased on coordination. It has been suggested that the ligands with nitrogen and oxygen donor systems inhibit enzyme activity. Coordination reduces the polarity of the metal ion mainly because of the partial sharing of its positive charge with the donor groups within the chelate ring system.(24,25).

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