

Synthesis and Characterization of some biologically active transition metal complexes for a ligand derived from dimedone with mixed ligands.

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Abstract:

The aim of the work is synthesis and characterization of bidentate ligand [3-(3-acetylphenylamino)-5,5-dimethylcyclohex-3-enone][HL], from the reaction of dimedone with 3-amino acetophenone to produce the ligand [HL], the reaction was carried out in dry benzene as a solvent under reflux. The prepared ligand [HL] 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 [HL] was used as a primary ligand while 8-hydroxy quinoline [HQ] was used as a secondary ligand with metal ion $\text{M}(\text{II})$. Where $\text{M}(\text{II}) = (\text{Mn}, \text{Co}, \text{Ni}, \text{Cu}, \text{Zn}, \text{Cd}$ and $\text{Pd})$ at reflux, using ethanol as a solvent, KOH as a base. Complexes of the composition $[\text{M}(\text{L})(\text{Q})]$ with (1:1:1) 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 tetrahedral geometry around ($\text{Mn}^{\text{II}}, \text{Co}^{\text{II}}, \text{Ni}^{\text{II}}, \text{Cu}^{\text{II}}, \text{Zn}^{\text{II}}$ and Cd^{II}) ions and square planer around (Pd^{II}) ion. The biological activity of the ligands [HL],[HQ] and complexes (MLQ) were studied using inhibition method.

Keywords: dimedone , 3-aminoacetophenone , β -enaminone , 8-hydroxy quinoline

1. Introduction:

The β -enaminones have gained a great deal of attention in organic synthesis due to their valuable role as precursors for synthesis of bioactive compounds such as anticonvulsants (acetyl choline –stersane inhibitor and oxytocin antagonists)[1-5].

These β -enaminones usually prepared from the reaction of aromatic and aliphatic amines with β -dicarbonyl compounds using catalyst [6-12].

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Mixed-ligand complexes plays an important role in numerous chemical and biological systems like water softening, ion exchange resin, electroplating, dying, antioxidant, photosynthesis in plants, removal of undesirable and harmful metals from living organisms. Many of these metals complexes shows good biological activity against pathogenic micro organisms [13-19]. The mixed ligand complexes are suitable for mimicking the role of metal ions, detoxification mechanism and drug designing. New mixed ligand complexes were continuously studied to get biologically active compounds [20]. In this work β -enaminone ligand [HL], [3-(3-acetylphenylamino)-5,5-dimethylcyclohex-3-enone] was prepared and used primary ligand together with 8-hydroxy quinoline [HQ] as secondary ligand. The complexes of these mixed ligands with some metal ions have been studied and characterized.

2. Experimental

2.1 Materials and Reagents

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

2.2 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 Conductivity. Elemental microanalyses were recorded by microanalysis (C.H.N) analyzer, Euro (Vector EA 3000A). Spectra for the ligand [HL] were recorded in DMSO- d^6 using Bruker, 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

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volume of 100ml with deionized water. Then, the metal content in the complexes were determined using atomic absorption spectroscopy [21]. The magnetic susceptibility of the complexes was obtained by using (Balance Johnson Matthey). The mass spectrum for the ligand [HL] was obtained by Electron-Impact (EI) on Shimadzu GCMSQPA 1000 spectrometer.

3. Synthesis

3.1. Synthesis of the ligand [HL]

Dimedone (1g, 7.13 mmole) was grinded together with 3-amino acetophenone (0.96 g, 7.10 mmole), then adding (25ml) of dry benzene. The mixture was allowed to continuous stirring until complete dissolution and formation of brownish light color solution. To the solution few drops of glacial acetic acid was added, then resulting solution was refluxed for (9) hrs until the light yellow precipitate was observed, the reaction was monitored by TLC. The reaction mixture was allowed to cool to room temperature, a light yellow product was isolated by filtration and washed several times with benzene and dried to give a yellow precipitate. Weight (1.42 g), yield (80 %), m.p (178-180) °C. The synthesis route of the ligand is shown in scheme (1). The microanalysis of results for the ligand and some of its physical properties is given in table (1).

3.2. Synthesis of the mixed-ligand [HL] and 8-hydroxy quinoline complexes with

some metal ions (MLQ).

(3.2.1) Synthesis of [Mn(L)(Q)](1)complex.

The metal solution of $MnCl_2 \cdot 4H_2O$ (0.15g, 0.75 m mole) in (10) ml ethanol was stirred for (10) minutes. The ligand solution (0.2 g, 0.77 mmole) in (10) ml ethanol after adjusted to pH=9 using few drops of KOH solution was added to the metal solution. Finally a solution of 8-hydroxy quinoline (0.11, 0.75 m mole) in (10) ml ethanol 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 ethanol and dried at room temperature during (24) hrs. A mustard solid was obtained. Weight (0.21 g), yield (72.41%), m.p (>350 °C) dec.

A similar method to that mentioned in preparation of (Mn) complexes was applied for the preparation of (Co^{II}), (Ni^{II}), (Cu^{II}), (Zn^{II}), (Cd^{II}) and (Pd^{II}) complexes. The physical properties and elemental mixed ligand (MLQ) analysis of complexes is given in table (2) below:

4. Results and Discussion

The synthesized ligand (3-(3-acetylphenylamino)-5,5-dimethylcyclohex-3-enone) [HL] has been characterized by FT-IR, ^1H , ^{13}C -NMR, Mass spectra, (C.H.N), UV-Vis spectroscopic methods.

4.1. NMR and IR spectra

The IR spectra of the free bidentate ligands show characteristic bands due to $\nu(\text{O-H})$, $\nu(\text{N-H})$, $\nu(\text{C=O})_{\text{acet.}}$, $\nu(\text{C=O})_{\text{di}}$, $\nu(\text{C=N})$ functional groups. The IR spectra of the complexes exhibited bands with appropriate shifts due to complex formation table (3). The absence of a peak around 3182 cm^{-1} and 3240 cm^{-1} in all complexes indicates deprotonation of (O-H) and (N-H) groups upon complex formation. The $\nu(\text{C=O})$ for acetophenone vibration at 1683 cm^{-1} in the free ligand [HL] is shifted and observed around $(1598-1639)\text{ cm}^{-1}$ for complexes indicating coordination of the oxygen atom of the acetophenone to the metal atoms[22], and the $\nu(\text{C=N})$ for free ligand 8-hydroxy quinoline vibration at 1579 cm^{-1} is shifted and observed around $(1498-1550)\text{ cm}^{-1}$, indicating coordination of the nitrogen atom of 8-hydroxy quinoline to the metal atoms[23]. At lower frequency the complexes exhibited new bands around $(467-588)\text{ cm}^{-1}$ and $(400-495)\text{ cm}^{-1}$ which are assigned to $\nu(\text{M-N})$ and $\nu(\text{M-O})$ vibration modes, respectively[24,25]. ^1H and ^{13}C -NMR spectrum of the ligand [HL] in DMSO- d_6 displayed signals corresponding to the various proton and carbon which confirms the structure of the ligand as shown in tables (4,5):

4.2. Mass spectrum

The mass spectrum of the ligand [HL] was also consistent with the proposed structure formula, (see section 3.1). The mass spectrum is displayed in fig.(1)

4.3. Electronic spectra, magnetic moments and conductivity measurements

The (U.V-Vis) spectrum for the ligand [HL], table (6), Fig.(2) exhibits two absorption peak at $(264)\text{ nm}$ $(37878)\text{ cm}^{-1}$ ($\epsilon_{\text{max}}=1325\text{ molar}^{-1}\text{ cm}^{-1}$) and $(309)\text{ nm}$ $(32362)\text{ cm}^{-1}$ ($\epsilon_{\text{max}}=1030\text{ molar}^{-1}\text{ cm}^{-1}$) which assigned to $(\pi\rightarrow\pi^*)$ and $(n\rightarrow\pi^*)$ transition[26]. The (U.V-Vis) spectrum for the 8-hydroxy quinoline [HQ], Fig.(3) exhibits have two absorption peaks, at $(273)\text{ nm}$ $(36630)\text{ cm}^{-1}$ ($\epsilon_{\text{max}}=520\text{ molar}^{-1}\text{ cm}^{-1}$) and $(316)\text{ nm}$ $(31645)\text{ cm}^{-1}$ ($\epsilon_{\text{max}}=467\text{ molar}^{-1}\text{ cm}^{-1}$) which were assigned to $(\pi\rightarrow\pi^*)$ and $(n\rightarrow\pi^*)$ transition respectively[27]. 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[28]. Thus (UV-Vis) spectrum of $[\text{Ni}(\text{L})(\text{Q})]$ complex, exhibits

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five peaks Fig.(4), the first, second and third high intense peaks at (258)nm (38759) cm^{-1} ($\epsilon_{\text{max}}=1250 \text{ molar}^{-1}\text{cm}^{-1}$), (268)nm (37313) cm^{-1} ($\epsilon_{\text{max}}=1380 \text{ molar}^{-1}\text{cm}^{-1}$) and (329)nm (30395) cm^{-1} ($\epsilon_{\text{max}}= 159 \text{ molar}^{-1}\text{cm}^{-1}$) are due to the ligand field. These peaks were shifted to lower frequency in comparison with the spectrum of the free ligands. The fourth peak at (341) nm (29325) cm^{-1} ($\epsilon_{\text{max}}=162 \text{ molar}^{-1}\text{cm}^{-1}$) is due to the charge transfer transition. The fifth peak at (406) nm (24630) cm^{-1} ($\epsilon_{\text{max}}= 162 \text{ molar}^{-1}\text{cm}^{-1}$) is due to the (d-d) electronic transition type (${}^3T_1 \rightarrow {}^3T_{1(P)}$) suggested distorted tetrahedral structure around the (Ni^{II}) ion [29] as shown in fig (2), where a comparison between the spectra of the mixed ligands and (Ni^{II}) metal ion is given.

These data together with the magnetic moment indicate tetrahedral geometry around the metal atoms studied except (Pd^{II}) atom being square planner, scheme (2). The molar conductivities indicate that all metal complexes are non-electrolytes [29]. Table (2)

5- 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:L:Q) as (1:1:1).

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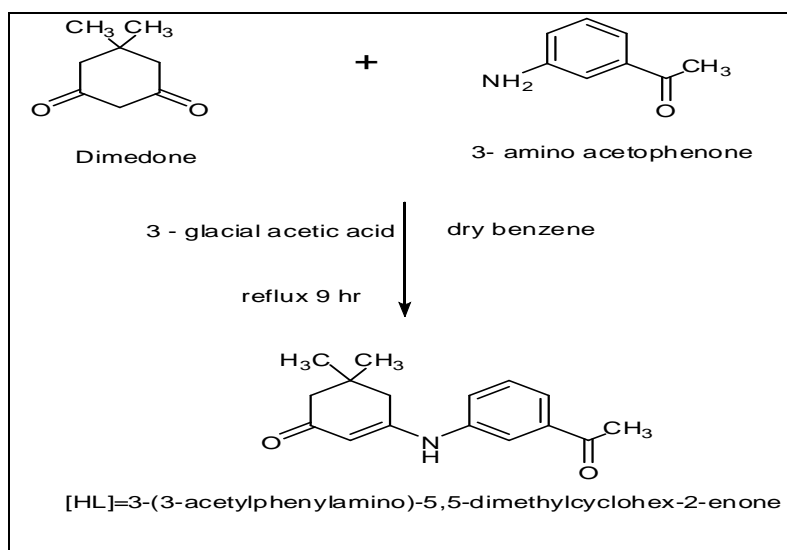
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Scheme (1) Synthesis route of the ligand [HL]

Table (1) Microanalysis results for the ligand and some physical properties for the ligand [HL].

Compound	Empirical formula	M.wt	Yield %	M.P C°	Colour	Found (calc.) %		
						C	H	N
[HL]	C ₁₆ H ₁₉ NO ₂	257.32	80	(178-180)	Pale yellow	74.55 (74.68)	7.28 (7.44)	5.35 (5.44)

M.P=melting point; Calc=calculated

Table (2) some physical properties of the prepared mixed- ligand complexes MLQ and their reactant quantity

No.	Empirical formula	Color	Dec.	Wt of metal salt (g)	Wt of product (g)	Yield %	Λ_m ohm ⁻¹ .cm ² mol ⁻¹
1	[Mn(L)(Q)]	Mustard	> 350	0.15	0.21	72.41	17.01
2	[Co(L)(Q)]	Dark green	>350	0.18	0.21	61.76	2.62
3	[Ni(L)(Q)]	Pale green	> 350	0.18	0.19	55.88	13.36
4	[Cu(L)(Q)]	Brown	> 350	0.13	0.25	71.42	1.42
5	[Zn(L)(Q)]	Pale yellow	> 350	0.10	0.18	52.94	0.17
6	[Cd(L)(Q)]	Olive	> 350	0.17	0.22	57.89	13.58
7	[Pd(L)(Q)]	Mustard	> 350	0.13	0.22	59.45	0.34

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Dec. =Decomposition

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

Compound	$\nu(\text{OH})$	$\nu(\text{NH})$	$\nu(\text{C=O})_{\text{acet.}}$ $\nu(\text{C=O})_{\text{di.}}$	$\nu(\text{C=N})$	$\nu(\text{C-N})$ $\nu(\text{C-O})$	$\nu(\text{M-N})$	$\nu(\text{M-O})$
[HL]	–	3240(s)	1683 (s) 1573 (s)		1242(s) –	–	–
8-Hedroxy quinoline	3182(br)	–	–	1579 (s)	– 1274(s)	–	–
[Mn(L)(Q)]	–	–	1604 (s) 1577 (s)	1550 (m)	1271(s) 1238(m)	588 (m) 467(m)	460(s) 403 (s)
[Co(L)(Q)]	–	–	1604 (s) 1577(s)	1498(m)	1282(s) 1226(m)	547(m) 501(s)	457(s) 410(s)
[Ni(L)(Q)]	–	–	1600 (s) 1577 (s)	1498(s)	1282(s) 1263(s)	547(w) 501(s)	457(m) 409(m)
[Cu(L)(Q)]	–	–	1598 (m) 1573 (s)	1498 (m)	1278 (s) 1222 (m)	582 (m) 520m)	430 (s) 403 (m)
[Zn(L)(Q)]	–	–	1604 (s) 1577(s)	1498 (m)	1271(s) 1240 (m)	505 (m) 484(s)	447 (s) 404 (s)
[Cd(L)(Q)]	–	–	1600(s) 1573(s)	1529(m)	1269(m) 1240(m)	503(s) 483(s)	495(s) 400(m)
[Pd(L)(Q)]	–	–	1639(w) 1571(w)	1498(s)	1282(s) 1215(s)	530 (w) 474(s)	416 (w) 408 (s)

br = broad, M=medium, S=Strong, W=weak

Table (4) $^1\text{H-NMR}$ data for [HL] measured in DMSO-d^6 and chemical shift in ppm (δ)

Compound	Funct. Group	δ (ppm)
[HL]	N-H [enamine group]	(8.9) (1H, s)
	Ar-H ($\text{C}_{10}, \text{C}_{11}, \text{C}_{12}, \text{C}_{14}$)	(7.7-7.3) (4H, m)
	C_2 for C_2H group	(5.3) (1H, s)
	C_{16} for CH_3 group	(2.6) (3H, s)
	DMSO solvent	(2.4)
	C_4, C_6 for CH_2 groups	(2.3) (4H, s)
	C_7, C_8 for CH_3 groups	(1.0) (6H, s)

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Table (5) ^{13}C -NMR data for [HL] measured in DMSO- d_6 and chemical shift in ppm (δ)

Compound	Funct. Group	δ (ppm)
[HL]	C=O _{acet.} for aromatic ring	197.5
	C=O _{di} for aliphatic ring	195.6
	C ₃ -NH group	159.6
	C ₉ for aromatic ring	139.7
	C ₁₃ for aromatic ring	137.7
	C ₁₁ for aromatic ring	129.1
	C ₁₀ for aromatic ring	127.4
	C ₁₂ for aromatic ring	124.1
	C ₁₄ for aromatic ring	121.4
	C ₂ for C ₂ H group	97.1
	C ₄ , C ₆ for CH ₂ groups	50.1
	DMSO solvent	40.3
	C ₅ for CH ₃ groups	32.2
	C ₇ , C ₈ for CH ₃ groups	27.8
C ₁₆ for CH ₃ group	26.7	

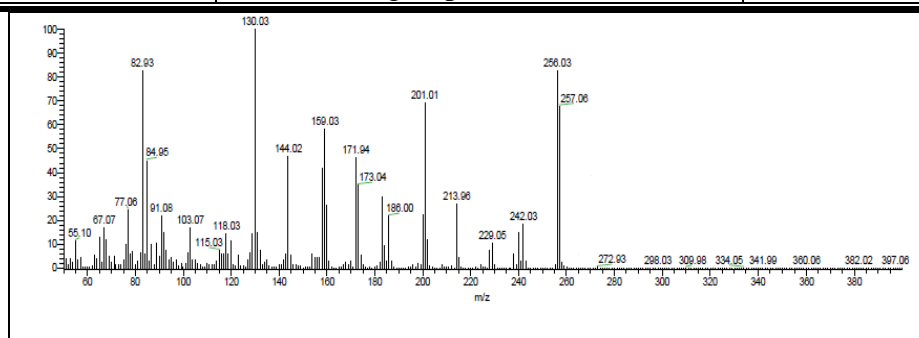
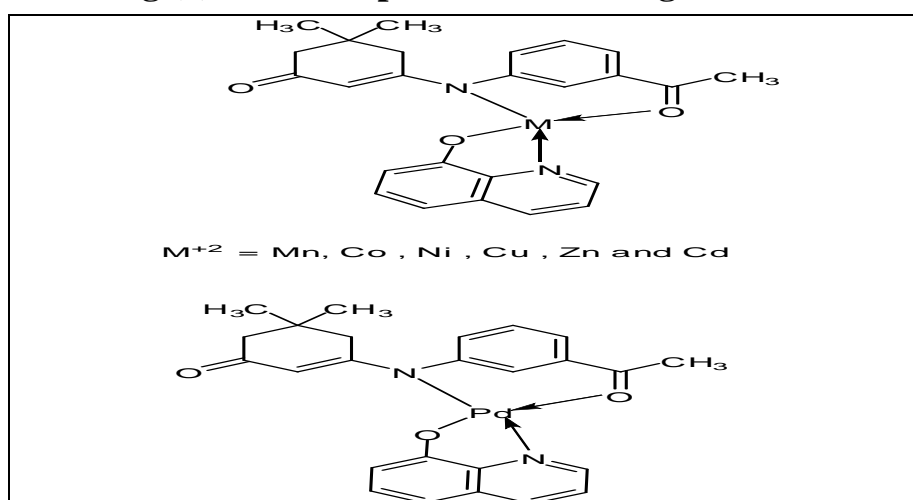


Fig.(1) EI-mass spectrum for the ligand [HL]



Scheme (2) A proposed structure of the complexes MLQ

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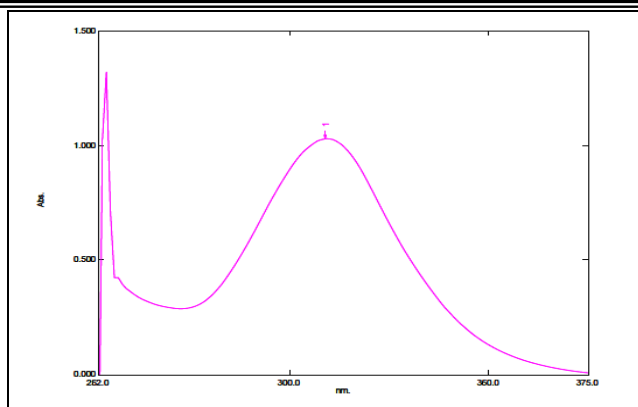


Fig.(2) Electronic spectrum of the ligand[HL]

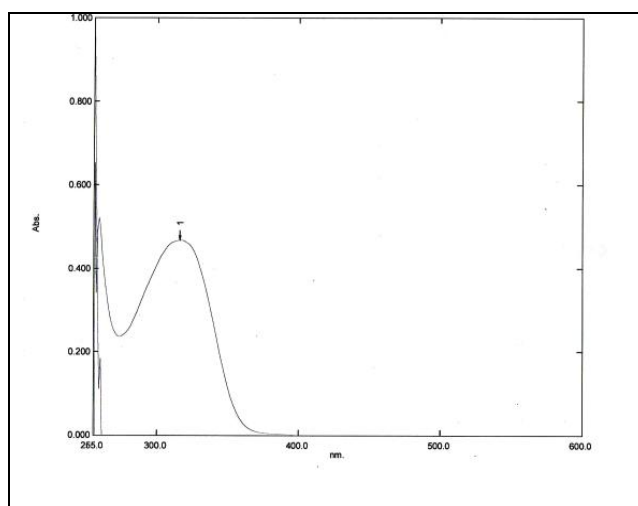


Fig. (3) Electronic spectrum of the ligand [Q]

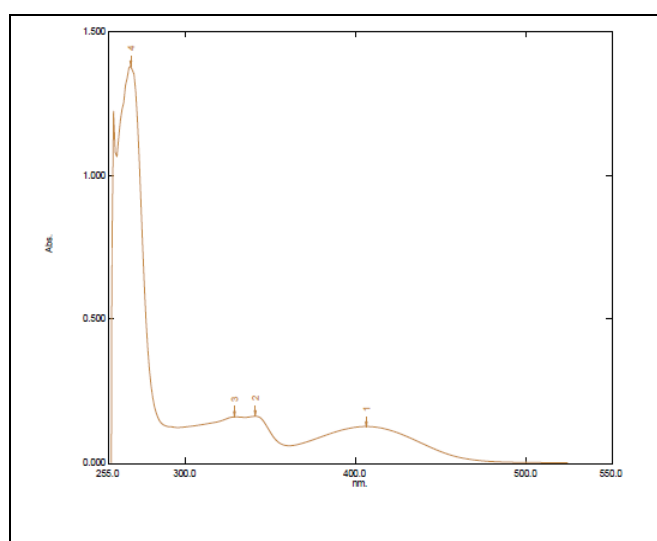


Fig.(4) Electronic spectrum of [Ni(L)(Q)]

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Table (6) Electronic spectral data of metal complexes MLQ

No.	Compound	μ_{eff} (BM)	Wave number		ϵ_{max} molar ⁻¹ cm ⁻¹	Assignment
			nm	Cm ⁻¹		
1	[Mn(L)(Q)]	5.87	260	38461	990	L.F
			265	37735	620	L.F
			292	34246	177	C.T
			381	26246	78	⁴ A ₁ → ⁴ E, ⁴ T _{1(G)}
2	[Co(L)(Q)]	4.70	257	38910	1992	L.F
			340	29411	97	C.T
			412	24271	125	⁴ A ₂ → ⁴ T _{1(P)}
3	[Ni(L)(Q)]	3.60	258	38759	1250	L.F
			268	37313	1380	L.F
			329	30395	159	C.T
			341	29325	162	C.T
			406	24630	126	³ T ₁ → ³ T _{1(P)}
4	[Cu(L)(Q)]	1.91	256	39062	1400	L.F
			258	38759	1126	L.F
			266	37593	1286	L.F
			340	29411	89	C.T
			398	25125	86	² T ₂ → ² E
5	[Zn(L)(Q)]	diamagnetic	256	39062	142	L.F
			259	38610	220	L.F
			308	32467	84	C.T
6	[Cd(L)(Q)]	diamagnetic	256	39062	1502	L.F
			258	38759	1650	L.F
			265	37735	1554	L.F
			324	30864	273	C.T
			404	24752	129	C.T
7	[Pd(L)(Q)]	diamagnetic	256	3406236	450	L.F
			271	900	289	L.F
			324	30864	23	C.T
			338	29585	23	¹ A _{1g} → ¹ E _g
			437	22883	26	¹ A _{1g} → ¹ B _{1g}

تحضير وتشخيص بعض معقدات العناصر الانتقالية النشطة حيويًا

مع مزيج لليكاند مشتق من الدايميدون ولليكاندات مختلطة

أ. د. أحمد ثابت نعمان أ. م. د. حسام حيدر الطويل

إيمان مطر عطيه

الخلاصة

أن الهدف من العمل تخليق وتشخيص ليكاند ثنائي السن

[3-(3-acetylphenylamino)-5,5-dimethylcyclohex-3-enone].[HL]

من تفاعل dimedone مع 3-aminoacetophenone لينتج الليكاند [HL], أُجري التفاعل باستخدام البنزين الجاف كمذيب وبطريقة التصعيد الحراري, تم تشخيص الليكاند المحضر باستخدام اطياف الاشعة تحت الحمراء واطياف الاشعة فوق البنفسجية وطيف الرنين النووي المغناطيسي للبروتون وللكاربون 13

وطيف الكتلة والتحليل الدقيق للعناصر ودرجة الانصهار. حضرت معقدات مزج الليكاند تم

استخدام الليكاند [HL] كليكاند اول بينما 8-hydroxy quinoline كليكاند ثاني مع M(II).

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

بطريقة التصعيد الحراري باستخدام الايثانول كمذيب ، هيدروكسيد البوتاسيوم كقاعدة،

بالصيغة [M(L)(Q)] بنسبة مولية (1:1:1). 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 رباعي السطوح حول الايونات الفلزية

(Mn^{II} , Co^{II} , Ni^{II} , Cu^{II} , Zn^{II} and Cd^{II}) والشكل الهندسي مربع مستوي حول الايون

الفلزي (Pd^{II}). تم دراسة الفعالية البيولوجية لليكاند [HL] والمعقدات MLQ باستخدام

طريقة التثبيط.