

Synthesis and Spectral Studies of Cr(III), Mn(II), Co(II), Ni(II), Cu(II) and Cd(II) Complexes with New Bidentate (N,O) Schiff Base Ligand bearing an Oxime functional group

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Abstract

The new Schiff base ligand has been prepared via condensation reaction of *o*-aminoacetophenone oxime and 3-Ethoxysalicylaldehyde. This ligand has been characterized by different techniques such as, (IR, U.V-Vis) spectroscopy, (¹H, ¹³C) NMR spectra, GC-Mass spectrum and micro-elemental analysis. Its Cr(III), Mn(II), Co(II), Ni(II) and Cu(II) complexes prepared and characterized by (IR, U.V-Vis) spectroscopy, magnetic susceptibility, conductivity measurements and atomic absorption. The ligand acts as monobasic bidentate with (N,O) set donor and coordinates through phenolic group oxygen atom and imine azomethine nitrogen atom. Octahedral geometry structure proposed to Cr(III), Mn(II), Cu(II) and Cd(II) show, while tetrahedral and square planar structures were proposed to complexes of Co(II) and Ni(II), respectively.

Keywords: bidentate (N,O) Schiff base, salicylidine complexes, *m*-aminoacetophenone oxime, 3-Ethoxy-salicylidine-*m*-aminoacetophenone oxime.

Introduction:

Schiff bases represent one of the most widely used families of organic compounds and their chemistry is essential material in many organic chemistry textbooks^(1,2). The easily preparation route of this kind and its ability of forming metal complexes with different metal ions makes chemists very interesting in synthesis and characterization of Schiff bases and their complexes^(3,4). Schiff bases have different chelation behavior, where it able to coordinate with metal ions as mono-⁽⁵⁾, bi-⁽⁶⁾, tri-^(3,4,7), tetra-⁽⁸⁾, penta-⁽⁹⁾, hexa⁽¹⁰⁾-, and heptadentate⁽¹¹⁾. Schiff bases complexes have various applications in catalytic field^(12,13). Also the (C=N) linkage is essential for biological activity, several azomethines were reported to possess remarkable antibacterial⁽¹⁴⁾, antifungal⁽¹⁵⁾, anticancer⁽¹⁶⁾,

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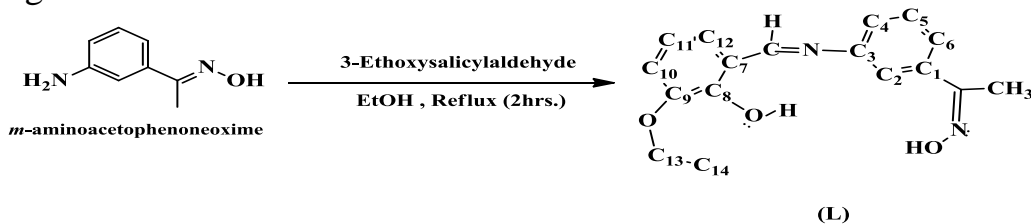
Experimental

Materials, Physical Measurements and analysis:

All reagents and solvents were obtained from commercial sources and used as received, except of precursor (*m*-AOX) was prepared as reported procedure⁽²¹⁾ with some modifications. Melting points were recorded by a Stuart melting point (digital) SMP30 apparatus. FTIR spectra were recorded by a Shimadzu (FT-IR) model 4800S spectrophotometer in the range (4000-400) cm⁻¹ by using KBr-disc. The U.V.-visible spectra of compounds were recorded by using a (U.V-Visible) spectrophotometer type Cary 100con. in the range (800–200) nm. GC-MS analysis of prepared ligand was performed on GC-MS QP-2010 (Shimadzu). ¹H and ¹³C NMR spectra of precursor and ligand were recorded in DMSO-d₆ by using a Bruker DMX-500 spectrophotometer (300 MHz). Elemental analyses of complexes were performed on an EA: Company: Euro Vector, Model: EA 3000A. Magnetic susceptibility of prepared complexes determined at (R.T) °C by Balance Magnetic Susceptibility, Model (MSB-Mk1). The percentages were determined by using a Phoenix-986 AA spectrophotometer. Conductivity measurements were recorded at (R.T) °C for solutions of samples in DMSO solvent using an Inolab Multi 740, WTW 82362 Weilhiem-germany.

Synthesis of 1-(3-((*E*)-(3-ethoxy-2-hydroxybenzylidene) amino)phenyl) ethanoneoxime (L)

The ligand (L) also prepared according to the previously mentioned procedure⁽²¹⁾. To stirred hot solution (1.66 g, 10 mmol) of 3-ethoxysalicylaldehyde in (15 ml) of absolute ethanol, (1.5 g, 10 mmol) of (*m*-AOX) in (15 ml) of hot absolute ethanol was added. The final reaction mixture refluxed to 4hrs. The beige precipitate which formed after slow evaporation of reaction mixture at room temperature, filtered off, dried and finally recrystallized from hot absolute ethanol. The purity of product tested with TLC technique by using (7.5 : 2.5) ml ratio of (benzene:ethylacetate).Scheme (1) represents the preparation reaction of ligand.



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Preparation of Complexes:

All chelation complexes were prepared by the same method, where (5 ml) warm ethanol solution of (0.5 mmol) metal chloride salts were added slowly to a stirred (15 ml) hot ethanol solution of (L) (0.298 g, 1 mmol). Color change has been noticed after mixing both solutions. The resulting solutions were then heated under reflux. All complexes precipitated after reduced the volume of reaction mixture by slow evaporation at room temperature, except Mn(II) and Cd(II) (B₂ and B₆) complexes were precipitated immediately after mixing both solutions. All obtained chelation complexes were filtered off, dried, washing with diethyl ether and distilled water (except B₁ complex) and finally recrystallized from hot absolute ethanol.

Results and discussion

Characterization of Ligand (L) :

¹H and ¹³C NMR Spectra:

The assignments of the chemical shifts in the (¹H and ¹³C) NMR spectra are listed in table (1).

¹H NMR spectrum of (L) in DMSO-d₆ exhibited signals at (δ=13.22 ppm, 1H), (δ=11.33 ppm, 1H) and (δ=2.20 ppm, 3H), which are assigned respectively to phenolic⁽¹⁴¹⁾ hydroxyl group, oxime^(22,23) hydroxyl group and (CH₃) group⁽²²⁾. The ethoxy group represents as triple signal at (δ=1.37-1.33 ppm, 3H) and quartet signal at (δ=4.085-4.063 ppm, 2H)^(24,25). The ¹³C NMR spectrum at same solvent exhibited chemical shifts at (δ=169.24 ppm)(δ=164.50 and 153.09) ppm were assigned to (C₈) atom and (C=N) of Schiff and oxime, respectively⁽²³⁾. The spectrum also revealed chemical shift at (δ=64.55 and 15.24) ppm were assigned to the carbon atoms (C₁₄ and C₁₃) of ethyl group, respectively⁽²⁴⁾. The chemical shift appeared at (δ=12.10 ppm) assigned to acetophenoneoxime methyl group^(22,23).

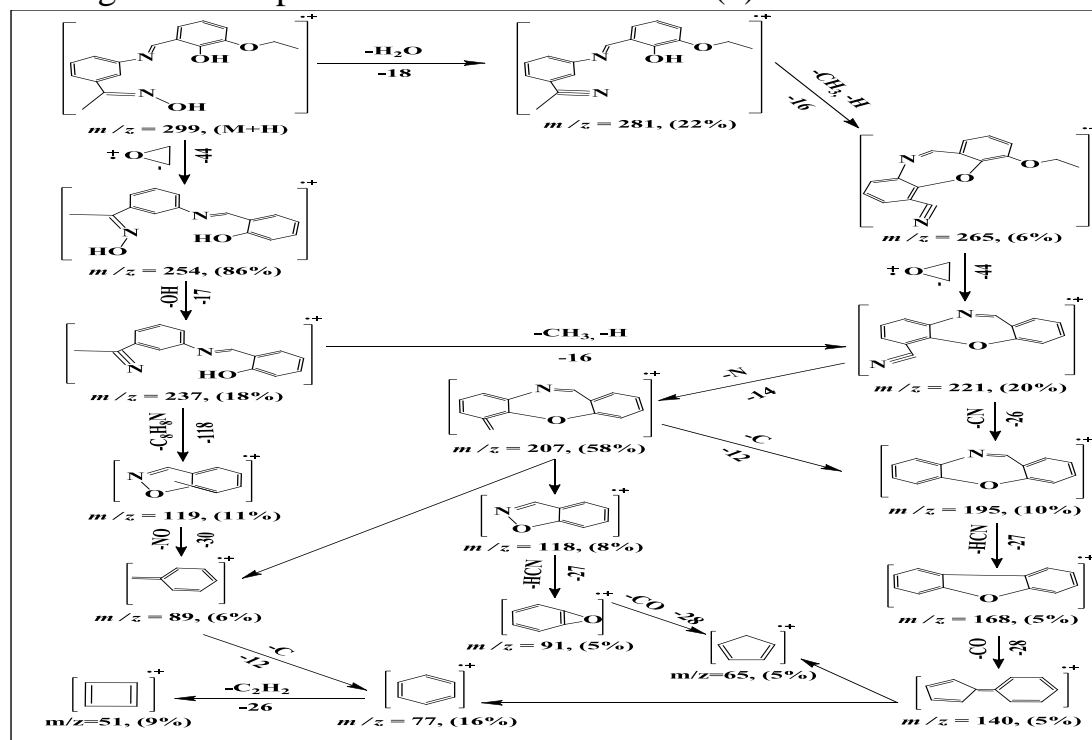
Table (1): (¹H and ¹³C) NMR spectrum data, chemical shifts assignment of (L) in DMSO-d₆

Compound Symbol	Assignment	Chemical shifts δ (ppm)
L	(phenolic, oxime)(OH) (CH=N, CH ₃)(H)	13.22 (1H,s) 11.33(1H,s) 8.99 (1H,s) 2.20 (3H,s)
	Aromatic rings protons	(6.86-7.63)(7H)
	(CH ₂)(H) (CH ₃)(H)	3.98-3.69(2H,q) 1.32-1.28(3H,t)
	(C ₈), (C=N), (C=N-OH), (CH ₃)	(169.24), (164.50), (153.09), (12.10)
	(C ₃ , C ₉ , C ₁ , C ₅ , C ₆ , C ₄ , C ₁₂ , C ₂ , C ₇ , C ₁₀ , C ₁₁), (C ₁₃ , C ₁₄)	(151.38, 148.48, 147.51, 138.80, 129.95, 124.56, 121.91, 119.76, 119.03, 117.45, 100.62,) (64.55, 15.24)

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GC-Mass spectrum of (L):

The mass spectrum analysis showed mother ion peak at ($m/z=299$), corresponds to ($M^+ + H$). The other fragments and their relative abundances and fragmentation pattern are shown in scheme (2).



Scheme (2): Proposed fragmentation pattern of (L)

Characterization of Metal Complexes:

Table (2) represents the physical property, yield percentage and elemental analysis of ligand (L) and its metal complexes.

Table (2): Physical properties, yield percentage and elemental analysis of (L) and its Complexes (B₁-B₆)

Comp. symbol	Molecular formula	Mwt g.mol ⁻¹	Color	M.P. (°C)	Elemental analysis(%) Found(calc.)			
					C	H	N	M
(L)	C ₁₇ H ₁₈ N ₂ O ₃	298.34	Beige	140-142	71.39 (68.44)	6.19 (6.08)	8.92 (9.39)	-----
B ₁	[Cr(L) ₂ (H ₂ O) ₂]Cl	718.15	Pale-Brown	310-312(d)	55.525 (56.812)	5.228 (5.291)	8.027 (7.797)	6.817 (7.240)
B ₂	[Mn(L) ₂ (H ₂ O) ₂].3H ₂ O	739.70	Light-Orange	252-253	55.132 (55.157)	5.907 (5.948)	7.610 (7.570)	7.350 (7.427)
B ₃	[Co(L) ₂]	653.59	Pale-Brown	190-194	61.707 (62.424)	5.370 (5.202)	8.260 (8.568)	8.666 (9.016)
B ₄	[Ni(L) ₂]	653.35	Ocher-Yellow	220-224	61.892 (62.447)	5.241 (5.203)	8.900 (8.571)	8.210 (8.982)
B ₅	[Cu(L) ₂ (H ₂ O) ₂].3H ₂ O	748.31	Yellowish-Brown	122-124	54.494 (54.522)	5.327 (5.879)	9.271 (7.483)	9.172 (8.492)
B ₆	[Cd(L) ₂ (H ₂ O) ₂]	743.11	Orange	262-265(d)	54.550 (54.904)	5.907 (5.113)	7.610 (7.535)	14.770 (15.126)

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¹H NMR spectrum of [Cd(L)₂(H₂O)₂] (B₆)

The singlet signal which observed at ($\delta=13.22$ ppm, 1H) in ¹HNMR spectrum of (L), assigned to phenolic hydroxyl group⁽²²⁾, disappeared in the ¹HNMR spectrum of [Cd(L)₂(H₂O)₂] (B₆), this mean the deprotonation have been occurred to phenolic hydroxyl group upon complexation, at the same time the proton of oxime hydroxyl group appearance approximately at the same position compared with that of free ligand, this confirm the non-participation of this group in coordination. Two singlet signals observed the first at ($\delta=8.990$ ppm, 1H) and the second at ($\delta=8.338$ ppm, 1H), represent the protons of azomethine group which appeared at ($\delta=8.992$ ppm, 1H)⁽²⁶⁾ in ligand spectrum. This proton shifted to upfield^(9,27) as a result of participation of azomethine nitrogen atom in coordination, which causes in increasing the electronic density around the carbon nucleus of this group. Further details are characterize in table (3)

Table (3): ¹HNMR spectral data (cm⁻¹) of (L) and [Cd(L)₂(H₂O)₂](B₆) complex

Compound symbol		(L)	[Cd(L) ₂ (H ₂ O) ₂]
Chemical shifts δ (ppm)	Phenolic(OH)	13.22 (1H,s)	-----
	(oxime)(OH)	11.33(1H,s)	11.22(2H,s)
	(CH=N)(H)	8.992 (1H,s)	8.990 (1H,s), 8.338 (1H,s)
	Aromatic(H)	(7.639-6.866)(7H)	(7.553-6.363)(14H)
	CH ₃ -C=N-OH	2.206 (3H,s)	2.179 (6H,s)
	(CH ₂)(H)	3.98-3.69(2H,q)	4.041(4H, q)
(CH ₃)(H)	1.32-1.28(3H,t)	1.350 (6H, t)	

FTIR Spectra:

Selected absorption bands of ligand and its complexes are given in Table (4). The medium band which observed in FTIR spectrum of free ligand at (3250 cm⁻¹) due to phenolic ν (O-H) vibration^(26,28) group, disappeared^(29,30) in all complexes spectra, as same time the ν (C-O) vibration which observed at (1253 cm⁻¹) in ligand spectrum shifted by (21-29) cm⁻¹ to a lower⁽³¹⁾ frequencies. These indicated the involvement of oxygen hydroxyl group in coordination with metal ions. The ν (C=N) vibration of Schiff base which observed at (1618 cm⁻¹)^(32,33) in ligand spectrum, sometimes shifted by (17-19) cm⁻¹ to a higher^(31,34,35) frequencies and other times shifted by (10-16) cm⁻¹ to a lower^(30,31,34,36) frequencies. These two different behaviors in addition to the observed changes in ν (C-N) vibration to a higher and to lower frequencies supported the participation of this group in coordination with metal ions.

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Table (4): FTIR spectral data (cm⁻¹) of (L) and its metal complexes

Compound	L	B ₁	B ₂	B ₃	B ₄	B ₅	B ₆
$\nu(O-H)Oxime$	3306(m)	3219(s)	3327(s)	3331(s)	3261(m) 3205(m)	Ov.	3524(s) 3419(s) Coord.(H ₂ O)
$\nu(O-H)Phenol$	3250(m)	-----	-----	-----	-----	-----	-----
$\nu(C=N)Schiff$	1618(s)	1602(s)	1635(s)	1635(w)	1608(s)	1608(s)	1637(s)
$\nu(C=N)Oxime$	1597(s)	1597(sh)	1602(sh)	1604(m)	1597(sh)	1595(sh)	1604(s)
$\nu(C-N)$	1313(m)	1313(w)	1301(w)	1307(w)	1317(m)	1316(sh)	1311(m)
$\nu(C-O)$	1253(s)	1228(s)	1234(s)	1232(s)	1226(s)	1228(s)	1224(s)
$\nu(N-O)$	1012(s)	1006(w)	1008(m)	1006(m)	1010(w)	1006(m)	1018(sh)
$\nu(M-O)$	-----	538(m)	526(w)	528(w)	536(w)	536(w)	515(w)
$\nu(M-N)$	-----	427(w)	425(w)	443(w)	414(w)	435(w)	424(m)

Ov=overlap, S=strong, W=weak, M=medium, Sh=shoulder, br=broad

Magnetic measurements and Electronic spectra:

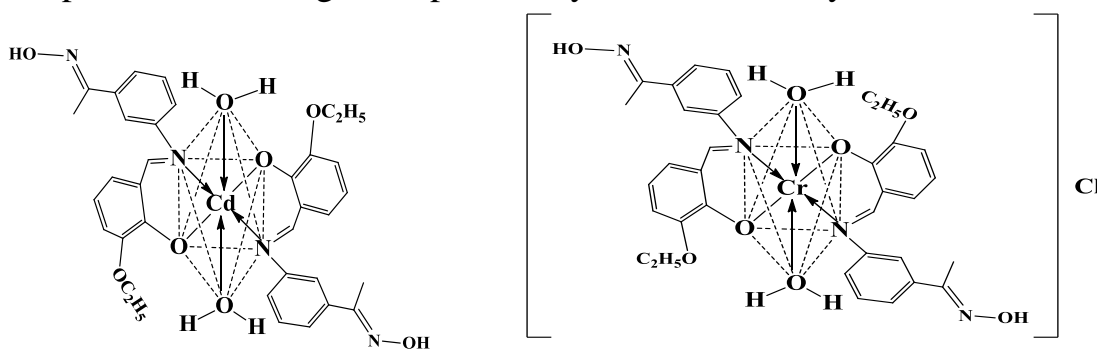
The spectral data and the magnetic moment of prepared complexes are listed in Table (5). The magnetic moment of the Cr (III) (B₁) complex has been found to be (3.84 B.M), which is at the range of an octahedral Cr (III) complexes⁽³⁷⁾. The electronic spectrum of (B₁) complex exhibited three absorption bands at (203, 233 and 287) nm which are respectively assigned to intraligand ($\pi \rightarrow \pi^*$) transitions⁽³⁸⁾. The spectrum also exhibited two absorption bands at (330 and 420) nm which are assigned respectively to ($n \rightarrow \pi^*$) transition of oximeazomethine group⁽³⁹⁾ and (MLCT) transitions⁽⁴⁰⁾. The calculated magnetic value of Mn(II) (B₂) was (5.84), which is in the range of octahedral high spin Mn(II) complexes⁽⁴¹⁾. The electronic spectrum of (B₂) shows new absorption band at (390 nm) due to (MLCT) transition⁽⁴²⁾. The magnetic moment of Co(II) (B₃) complex was (4.3 B.M) which is in the range of high spin d⁷ tetrahedral geometry⁽⁴³⁾. The electronic spectrum of (B₃) exhibited new absorption bands at (625 and 410) nm which are assigned respectively to (${}^4A_{2(F)} \rightarrow {}^4T_{1(P)}$) (ν_3) transition^(5,29,30) and (LMCT)^(29,30). The magnetic moment of Ni(II) (B₄) complex was (0.00 B.M) this confirm the square planar geometry of this complex. The absorption band related to ($n \rightarrow \pi^*$) transitions which observed in U.V-Vis spectra of free ligand at (353 nm) absent in spectrum of (B₄) complexes. This explained on the bases of stabilizes of non-bonding lone pair of azomethine group nitrogen atom as a result of donation of this non-bonding lone pair to metal ion through the coordination⁽⁴⁴⁾. Only (MLCT)^(45,46) transition has been observed in electronic spectrum of (B₄) complex. The magnetic moment of Cu(II) (B₅) complex was (1.94 B.M) this confirm the

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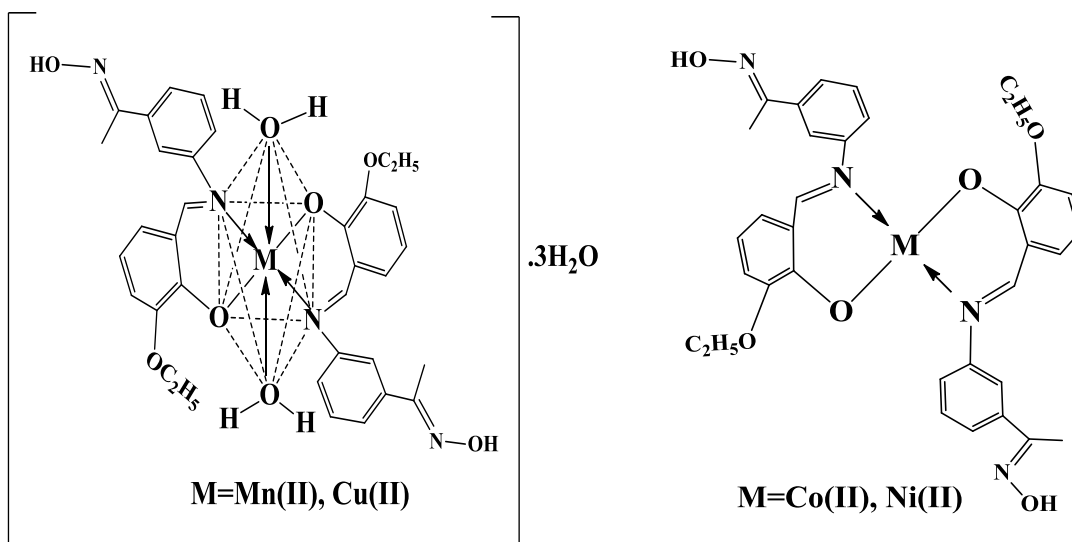
presence of one unpaired electron in octahedral geometry. Only (MLCT)^(47,48) transition has been observed at (398 nm) in electronic spectrum of (B₅) complex. The electronic spectrum of Cd(II) (B₆) complex in DMSO exhibited new absorption band at (399 nm) due to (MLCT)⁽⁴⁹⁾.

Conductivity Measurements:

The value of molar conductance of Cr(III) complex in DMSO was (29.10 S.cm² .mole⁻¹), indicated the 1:1 ratio electrolyte nature⁽⁵⁰⁾. The values of the other complexes (A₂) to (A₅) in DMSO were within the range (8.5-14.10S.cm² .mole⁻¹), indicated the non-electrolyte nature⁽⁵⁰⁾ of these complexes. According to all previously mentioned analyses we can



proposed the following structures of prepared complexes.



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Table (5): Electronic spectral data of (L) metal complexes

Compound symbol	$\lambda_{max} nm (\bar{\nu} cm^{-1})$	Assignment	$\mu_{eff} (B.M)$ Geometry
L	204 (49020), 228 (43860), 268 (37313)	$\pi \rightarrow \pi^*$	
	310 (32258), 353 (28329)	$n \rightarrow \pi^*$	
B ₁	203 (49261), 233 (42918), 287 (34843)	Intraligand ($\pi \rightarrow \pi^*$)	(3.84) O.h
	330 (30303)	($n \rightarrow \pi^*$)	
	420 (23810)	(LMCT)	
B ₂	242 (41322), 292 (34247)	Intraligand ($\pi \rightarrow \pi^*$)	(5.84) O.h
	390 (25641)	(LMCT)	
B ₃	203 (49261), 232 (43103), 272 (36765)	Intraligand ($\pi \rightarrow \pi^*$)	(4.3) T.h
	304 (32895)	($n \rightarrow \pi^*$)	
	410 (24390)	(MLCT)	
	625 (16000)	${}^4A_2^{(F)} \rightarrow {}^4T_1^{(P)} (\nu_3)$	
B ₄	202 (49505), 247 (40486)	Intraligand ($\pi \rightarrow \pi^*$)	(Dia.) S.P
	335 (29851)	($n \rightarrow \pi^*$)	
	400 (25000)	(MLCT)	
B ₅	206 (48544), 235 (42553)	Intraligand ($\pi \rightarrow \pi^*$)	(1.94) O.h
	290 (34483)	($n \rightarrow \pi^*$)	
	398 (25126)	(MLCT)	
B ₆	259 (38610)	Intraligand ($\pi \rightarrow \pi^*$)	Dia. O.h
	399 (25063)	(MLCT)	

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الخلاصة

تضمن البحث تحضير ليكاند شف جديد وذلك من مفاعلة المادة الاساس ميتا-اوكسيم مع 3-ايثوكسي سالسالديهايد. تم تشخيص الليكاند بالاعتماد على تقنيات مختلفه مثل مطيافيه الاشعه تحت الحمراء والاشعه فوق البنفسجيه- المرئيه والرنين النووي المغناطيسي والتحليل الدقيق للعناصر ومطيافية الكتله. اما المعقدات المحضره فقد تم تشخيصها اعتمادا على مطيافية الاشعه فوق البنفسجيه-المرئيه والتحليل الدقيق للعناصر والامتصاص الذري اللهبى وقياس الحساسيه المغناطيسيه والتوصيل المولاري. طيف الرنين النووي المغناطيسي استعمل في تشخيص معقد الكادميوم. اثبتت نتائج التحاليل ان الليكاند تتاسق مع الايونات الفلزيه بشكل ليكاند ثنائي المخلب من خلال ذرة اوكسجين مجموعة الهيدروكسيل العائده لحلقه الفينول وذرة نايتروجين مجموعة الازوميثين العائده لقاعدة شف. اعتمادا على نتائج التحاليل المستحصله تم اقتراح بنيه ثماني السطوح لمعقدات الكروم (III) والمنغنيز (II) والنحاس (II) , وقد تم اقتراح بنيتي رباعي السطوح والمربع المستوي لمعقدي الكوبلت (II) و النيكل (II) , وعلى التوالي.