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Synthesis and Characterization of two Closed Side Oxadiazole ligands with their Related Complexes Cu(II) and Pd(II)

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ABSTRACT

The two oxadiazole new ligands of bis oxadiazol derivatives, have been synthesized by the reaction of $\bar{1}, \bar{2}$ - bis -[$\bar{\bar{5}}, \bar{\bar{2}}$ Chloroethylsulphide)]1, 3, 4 - oxadaiazol - 2yl]ethane (L1)and $\bar{1}, \bar{2}$ - bis -[$\bar{\bar{5}}, \bar{\bar{2}}$ Chlororthyl sulphide)]، oxadiazol-2yl] methane (L2.) with adibic acid in presence of ethnolic solution of potassium hydroxide. The mixture was refluxed for eight hours. The ligands were characterized using IR 1H.NMR13C-NMR and mass spectroscopies .The (L3) was used to synthesize complexes with metals Cu(II) and pd(II) . The complexes were studied and identified using IR, UV-VIS spectra and molar conductivity, magnetic susceptibility and atomic absorption techniques. The results exhibited that all complexes have an octahedral structure.

Introduction

The attention of inorganic chemistry has been caught by the extraordinary of the co-ordination chemistry of metals in biological system [1]. The challenge of synthesizing model ligands which will be proved to those in the binding of the cooper proteins has been taken up and many new ligands have been produced for this purpose [2].

Depending on the fact that the compounds containing - SH group can be easily substituted [3] and the current interest in hetrocyclic-bis-oxadizole derivatives which reffered to fact that these compound have posse , pesticide insecticide, fungicidal and bacterial activity further the substituted axadituted were as antimitotic, muscle relaxants and tranquilizing agent [4]. Each of these closed ligands contains a thioether sulfur and oxygen and hetrocyclic nitrogen as donor atoms, particularly no work has been done in this field with complexes of these close macrocyclic ligands.

Prompted by these observations we aimed to obtain two closed side oxadiazole ligands and the characterization study of the complexes Cu(II) and Pd(II) with (L3) .

Experimental part:

A-preparation of ligands:

1- Preparation of 1, 2 – bis (5 (2 – chloroethylsulphide) – 1,3,4 – oxadizol – 2 yl) ethane (L_1)

To a solution of (1.2 gm , 4.65 mmol) of 1, 2 – bis (thiol 1,3,4 – oxadizol –2 yl) ethane in (15:10) ml (ethanol : water) was added to a solution of (0.53 gm , 9.46 mmol) of potassium hydroxide in ml of ethanol . The mixture was refluxed for 30 minutes , and left for five minutes , followed by addition to a beaker containing of ice which acidified with 5 ml of concentrated hydrohloric acid to give white precipitate compound which was filtered , recrystallized from ethanol to give white crystal with m.p (164 – 166 C°) and yield (78.35%) it soluble in ethanol , methanol , DMSO , chloroform.

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2- Preparation of 1, 2–bis (5(2– chloroethylsulphide) – 1,3,4 – oxadizol – 2 yl) methane (L_2)

To a 10 ml of ethanolic solution of (0.53 gm , 9.46 mmol) of potassium hydroxide was poured to a 25ml ethanolic solution of 1.035 gm , 4.24 mmol) of 1, 2 – bis (thiol 1,3,4 – oxadizol –2 yl) methane. The mixture was refluxed for 30 minutes , followed by addition of (0.83 gm , 0.67 mmol) of 1,2 dichloroethane and the refluation was obtained at laboratory temperature for 24 hours . The final mixture was evaporated to give pale – yellow crystal recrystallized from ethanol , dried under vacuum (m.p = 263 C°) , (yield = 76.28%) , soluble in ethanol , methanol , DMSO , and DMF .

3- Preparation of 1, $\bar{2}$ – di[2 thiolethyl {$1^{=}$,$4^{=}$ bis(1,3,4- oxadizol – 2 yl) ethane di adipate(L_3):

A 0.112 gm (2m.mole) of potassium hydroxide was dissolved in 5 ml of absolute ethanol then poured to a solution of 0.356 g (1 m.mole) of $\bar{1},\bar{2}$ - bis -[$\bar{\bar{5}}$, $\bar{\bar{2}}$(Chloroethylsulphide)] -1,3,4-oxadiazol-2yl] ethane (L_1) in 15 ml of ethanol , followed by addition of 15 ml of ethanol. The mixture was refluxed for 30 minutes. Then (10 ml) of ethanolic solution of 0.146 gm (1 m.mole) adibic acid was added dropwise to the mixture. The mixture was refluxed for 8 hours. The mixture was allowed to cooling and a white product was formed which was filtered and dried using rotatory evaporator, the product was recrystallized from ethanol and dried under vacuum (m.p =246C°d yield =83%), Table(1).

4- Preparation of 1,$\bar{2}$ di[2 thiolethyl {$1^{=}$,$4^{=}$ bis(1,3,4- oxadizol – 2 yl)}methane diadipate (L_4):

To a 15 ml of ethanolic solution of 0.332 gm (1 m.mole) of $\bar{1},\bar{2}$ - bis -[$\bar{\bar{5}}$, $\bar{\bar{2}}$(Chloroethylsulphide)] -1,3,4,- oxadiazol –2yl] methane (L_2) , a 0.112 gm (2 m.mole) of ethanolic solution of potassium hydroxide was added. The mixture was refluxed for 30 minuets . Then 0.146, gm (1.0 m.mole) of adibic acid was poured dropwise to the mixture. Then it refluxed for 8 hrs then it was left to cool at room temp. , a white product was precipitated, filtered and dried under vacuum , then recrystellized from ethanol (m.p = 246 C°d yield = 80.30%) Table(1).

B-Synthesis of complexes :

A –Preparation of [Cu (L_3)$_2$ Cl_2]

To an ethanolic solution of 0.0428 gm (0.1 m.mole) of (L_3). a 0.170gm (0.1m.mole) of $CuCl_2$ was added. The mixture was refluxed for 2 hrs then a red formation precipitat was filtered off , washed with hot ethanol, and dried formula and physical properties are listed in Table (1).

B - Preparation of [Pd (L_3)$_2$ Cl_2].

A 0.0172 gm (0.1 m.mole) of PdCl2 was dissolved in 5 ml of ethanol, then added to a 0.0428 gm (0.1 m.mol) of ethanolic solution of (L_3). The mixture was refluxed for 2 hrs, it was filtered , washed with hot ethanol to give red brown powder Table (1).

Results and discussion

Synthesis and characterization of (L_3) and (L_4)

The two ligands were synthesized according to scheme: (1)

These two closed macro ligand contained six oxygen atoms two sulfer atoms and four nitrogen atoms were characterized such as white crystals which are stable in a wide range of temperatures and soluble in some of organic solvent ethanol, methanol DMSO .

1- IR spectra:

Fig (1) and Table (2) showed the IR spectra of ligands(L_1-L_3), The IR spectra of the two ligands(L_3,L_4) exhibited the bands at (2923 ,2945) cm^{-}

^{1} which can be attributed to asym υ(C-H) ,sym υ (C-H) aliphatic and υ (O-CH_2) [5] respectively .The appearance of sharp absorption bands , one of which appearing at 1739 cm^{-1} and the other observed at (1695-1705) cm^{-1} were assigned to C=O stretching frequency corresponding to acyl carbonyl[6-8]

The appearance of bands which ranged between (1465-1455) cm^{-1} which assigned to υ(SH-CH_2) , while the disappearance of bands around (1256-1253)cm^{-1} which related to (CH_2-Cl) of starting compound (L_1) and (L_2) , indicating the replacing of Cl groups by menses of –CH_2-O group [9-10]

The bands which appeared around 1739 cm^{-1} may be due to the ester group [11]. The two ligands (L_3) and (L_4) exhibited bands with some shifting to the lower frequency at range (1645-1682) cm^{-1}, (1374-1303) cm^{-1}, (940-969) cm^{-1} and (1049 - 695) cm^{-1} respectively. These were due to υ(C=S +C=N), υ(C=C + C-S), υ (N-C-S) and υ(C-S) respectively [12, 13]

The bands around (1049-1062) cm^{-1} in the(L_3) and (L_4) are due to υ(C-O-C), which is usually used to identify the oxadiazol ring [13]

2- ^{1}H-N.M.R spectra:

The ^{1}H-n.m.r were recorded at 400 MHZ by using a MeOD solvent Fig (2) and Table (3)

The ^{1}H-n.m.r spectra showed principal signals , the signals at δ(2.5 – 2.6) ppm for ligand (L_3) may be due (CH_2) (2H) while the band at δ(4.28) ppm may be due to (q,2H,CH_2) [11] and δ(3.6) which assigned to (2H,-OCH_2) [14] for the tow mentioned ligands.

3- ^{13}C-N.M.R spectra:

^{13}C-n.m.r of the (L_3) and (L_4) which was carried out in (MeOD) Table (4) , Fig (3) and Fig (4) showed a set of signals at [δ=33.05, 38.96 ,39.30, 39.97, 40.30, 40.63, 40.97] ppm may be due to $^{-}C_4$ - (S-CH2) , while the signals at [δ=(14.34) and δ =61.79] ppm for L_3 may be due to $^{-}C_7$ -(O-CH_2) [15].

The signals which appeared at [δ = 129.12, 131.95, 132.15] ppm and 131.942, 128.294, 127, 22] ppm may be due to C'_3, C'_9 for (L_3) and (L_4) respectively [16]. While the signal at [δ = 167.38] ppm may be due to C'_1 oxadiazole ring [16]. A very important evidence of the appearance of C=O in the two synthesized compounds (L_3) and (L_4) was done by the ^{13}C-DEPT[17] (Distortions Enhancement by Polarization Transfer), which is used to determine the multiplicity of ^{13}C-signal the resulting ^{13}C-DEPT spectra containing only signals arising for protonated carbon non-protonated carbon did not give signals in the ^{13}C-DEPT . The signals arising in CH_3 and CH group showed positively phased from those in CH_2 groups. So signals from CH_3 and CH groups upwards (+) while signals for CH_2 groups point downwards (-). Here in ligand (L_4) and under the effect of resonance from there are appearance of signals at (10.12 and 14.34) ppm may due to C' -9(+) while the signals at (61.97) ppm appeared as (-) which was assigned to (C'_8-CH_2) carbon.

4- Mass spectra of L_3:

The mass spectra of the (L_3), scheme(2), is shown in the following pathway of fragmentation exhibited the parent ion at MZ = 428 with an abundance of (2.2%), which indicated that the parent molecule is unstable in this condition. Also the spectrum exhibited a number of peaks at [MZ = 427, 381, 371, 342, 304, 263, 242, 190, 141 and 100].

5- [Cu (L_3)$_2$ Cl_2], [Pd (L_3)$_2$ Cl_2]complexes:

The solid complexes of the(L_3) which [M = $Pd^{(II)}$ and $Cu^{(II)}$] were synthesized and in the molar ratio (2:1) (L:M) Fig (9). The ligand seems likely to coordinate via N-oxadiazole ring and sulfur sites with metal ion Table (2) Fig (5), Fig (6) and this was confirmed by C-N, C-S stretching frequencies. The complexes exhibited band of medium intensity in the region of 1550 cm^{-1} which is absente in the free ligand, this was assigned to the coordinated C-N vibration [16], the coordination of nitrogen with palladium metal ion [17].

The band at 880 cm^{-1} with shifting about(90) cm^{-1}[18] due to stretching vibration of υ(N-C-S), another strong sharp band at 1028 cm^{-1} due to υ(C-S) ,this fact was confirmed by the appearance of the medium band with shifting to lower frequency at 641 cm^{-1} and this indicated to the coordination of sulfur with palladium ion [19, 20] .The IR spectra showed a medium band at 419 cm^{-1} which was due to υ(Pd-N)[21, 22].

The comparsion of the IR spectra of the copper complexes with(L_3) showed The shifting of bands of υ (C=S + C=N) to the lower frequency at 1600 cm^{-1} with the change in intensity of the bonding of N with the metal ion. Also the lowering in frequency about (100 cm^{-1}) of υ(SCH_2) bond of copper complex compared with that in free ligand and the appearance of a sharp band at 1000 cm^{-1} was due to υ (N-C-S), this indicated binding of N and S with the Cu ion [22,23] .

The IR spectra showed two splitted bands at 762 cm^{-1} which refer to bonded sulfur . The band around 500 cm^{-1} may be due to υ (Cu-N) the far IR spectrum showed a band at 350 cm^{-1} which was due to υ(Cu-Cl)[25]

The UV – VIS. spectra data of (L_3) complexes are listed in Table (5). The (L_3) exhibited one strong band at 42553 cm^{-1} which was due to $\pi - \pi^*$ Fig (7).

The dark brown complexes of copper complexes Fig(8) showed bands at 30674 cm^{-1} 28169 cm^{-1} and calculated band (15917 cm^{-1}) expected for transition $^2Eg \rightarrow ^2T_2g$.[26]

The complex have suggested that the L_3 complex with copper ion was octahedral Fig(9). The molar conductivity estimated was measured in diethyl sulphoxide at 10^{-3}M which indicated non-electrolyte[27].

The atomic absorption suggested that the ratio of M: L was (6.6mg, 3.3mg) using equation curve fitting method Table (6), this is approximately in agreement to ratio (2:1). (L: M)

The UV-Vis. Spectra data of L_3 complex with palladium are listed in Table (5). The dark red brown crystal solution of Pd(II) complex in DMSO showed at 30648cm^{-1} was due to $^3A_{2g(F)} \rightarrow ^3T_{2g(F)}$ transition[28] . The conductivity of the palladium complex was estimated by their molar conductivity Table (5) in DMSO at 10^{-3} M indicate non-electrolyte[28]. The magnetic susceptibilities measured showed that the complexes have paramagnetic properties [28] Table (5).

Our investigation suggested that (L_3) complex with palladium metal ion was octahedral the structure of palladium complex with (L_3) is shown in Fig (9).

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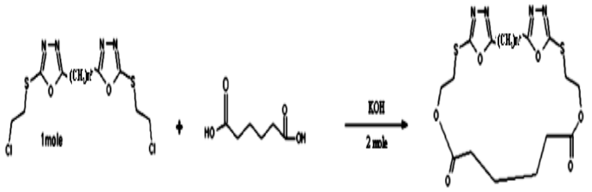
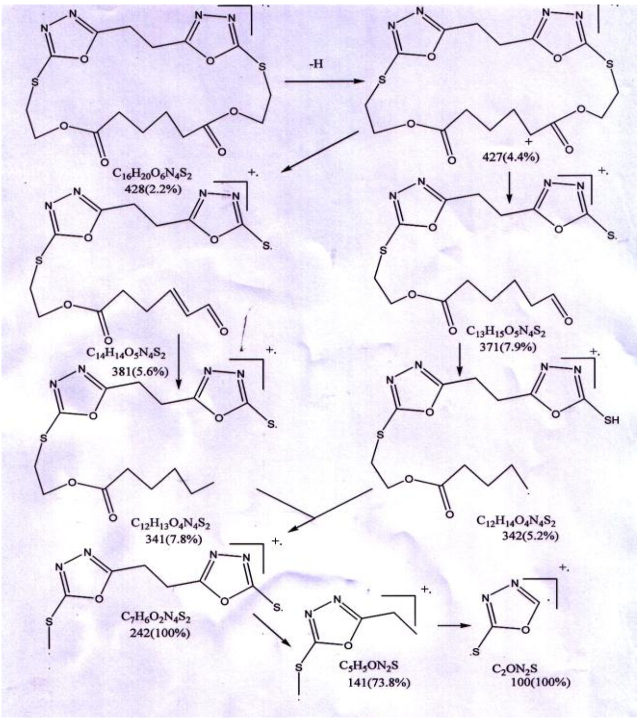


L_1; n = 2 L_3; n = 2
L_2; n = 1 L_4; n =1

Scheme (1)



Scheme (2), the fragments observed in the mass spectrum of (L3)

Table (1): The physical properties for prepared compounds

Compounds No.	Formula	Yield %	M.p C°d	Color	M.wt
L_3	$C_{16}H_{20}N_4S_2O_6$	83%	264	Pale-yellow	428
L_4	$C_{15}H_{18}N_4S_2O_6$	86%	244	Pale-yellow	414
$[Cu(L_3)_2Cl]$	$[Cu(C_{16}H_{20}N_4S_2O_6)_2Cl_2]$	80.30%	238	Dark-brwn	919.5
$[pd(L_3)_2Cl]$	$[Pd(C_{16}H_{20}N_4S_2O_6)_2Cl_2]$	81%	236	Dark red-brown	874

Table (2): IR spectra bands in $cm^{-1}$of the synthesized compounds

Compounds	ν(OH)	ν(NH)	ν(C-H) aleph	ν(S-H)	ν(C=S)(C=N)	ν(S-CH2)	ν(CH-Cl)	ν(C-O-C)	ν(N-C-S)	ν(C-N)	ν(M_N)	ν(M_S)
L_1	---	---	2922sym 3064 asym	---	$1483_{(s)}$	(1210_1404) (m)	1256(s)	1064 (m)	949(m)	---	---	---
L_2	---	3053	2846(sym) 2945(asym)	---	1482(m)	1408(m)	1253(m)	1042(s)	942-950	---	---	---
L_3	---	---	2923(m)	---	1374	1465	---	1049	940	---	---	---
L_4	---	---	2945(m)	---	1303	1455	---	1062	969	---	---	---
$[Cu(L_3)_2Cl_2]$	3500(b)	---	2910(w)	---	1650	1347	---	1047	880	1550	419	762
$[Pd(L_3)_2Cl_2]$	3500(b)	---	2900(w)	---	1600	1368	---	1025	850	1500	420	762

Table (3): The ^{1}H.N.M.R spectra band in ppm for synthesized compounds

Compounds	δ(2H) CH_2	δ (q,2H,CH_2)	δ (2H,O-CH_2)
L_1	2.5	…….	--------
L_2	3.5_3.6	……..	--------
L3	(2.5 _ 2.6)	4.28	3.6
L_4	2.6	4.4	3.4

Table (4): the 13C-N.M.R data of synthesized ligand

Compound	C_1^{-}	C_2^{-}	C_3^{-}	C_4^{-}	C_5^{-}	$^{-}C_6$	C_7^{-}	$^{-}C_8$	$^{-}C_9$
L_3	167.38	-----	129.12	33.05	-----	----	-----	61.79	131.942
			131.95	38.96					128.294

			L_4				
			167.38				

			-----				132.15
			-----		40.30	39.79	39.30
			-----				-----
	----		-----				
	----		------				----
	12.98		-----				14.34
10.12	127.322	128.294	131.942	14.34			127.77

Table (5): the characterization data of the synthesized compounds

No.	Compound	Color state	Uv-Vis cm^{-1} in DMSO	Transition	Mole conductivity $\Omega^{-1}\,cm^2\,mol^{-1}$	Magnecity suseaotibility B.M
1	(L_3)	Pale-yellow cryst	42553	$\pi - \pi^*$	2.04	---
2	(L_4)	Pale - yellow cryst	---	---	2.04	---
3	$[Cu(L_3)_2Cl_2]$	Dark - red crys.	30674(υ_2), 28169(υ_3), 15917(υ_1) (cal)	$^2Eg \rightarrow ^2T_{2g}$	2.04	1.66
4	$[Pd(L_3)_2Cl_2]$	Dark-red brown crys.	30671(υ_2), 27624(υ_3), 163633(υ_1)(Cal)	$^4A_{2g(F)} \rightarrow ^4T_{2g(}$	2.04	2.12

Table (6): The atomic absorption data for ligand $[Cu(L^3)_2Cl_2]$ complex.

Cu(II) conc. Ppm	Abs.
2	5
4	11
6	14
8	18
$(C_{16}H_{20}N_4O_6S_2)_2$ Cu	8

a = 1.5
b = 2.1
r = 0.9899
Conc. Of Cu(II) in the complex = 3.33 ppm
Conc. Of ligands = 1.00-3.33=6.66
L/M = 2/1

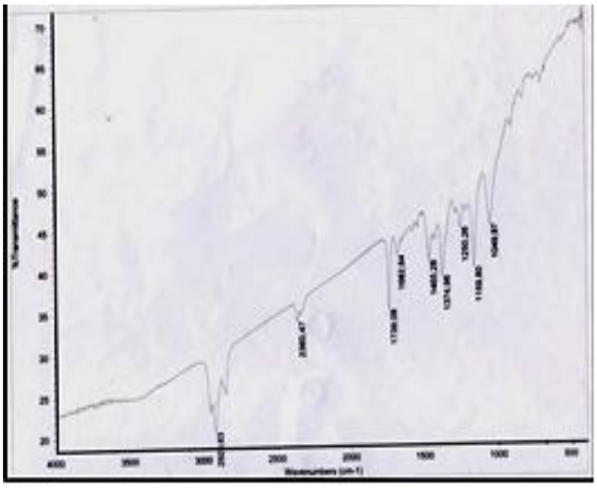


Fig (1): The IR spectrum of (L_3)

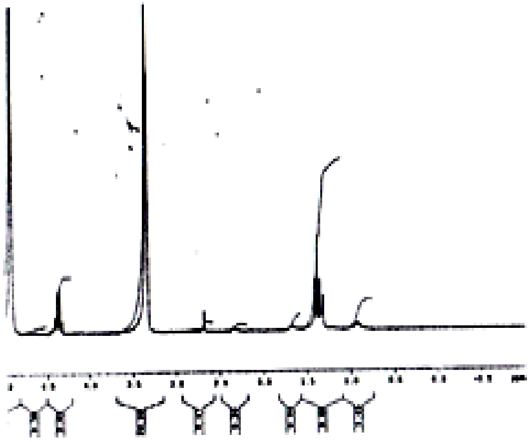


Fig (2): The ^{1}H-N.M.R spectra of(L_3)

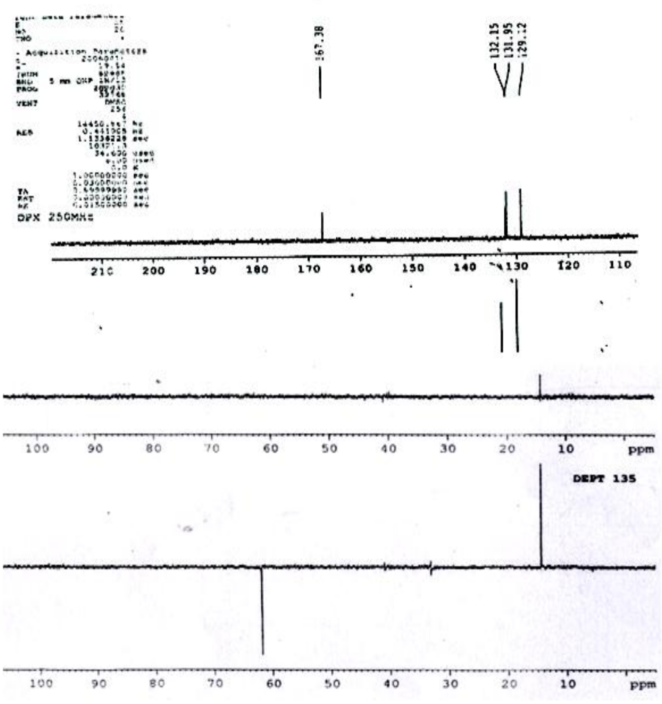


Fig (3): The 13C-N.M.R of (L3)

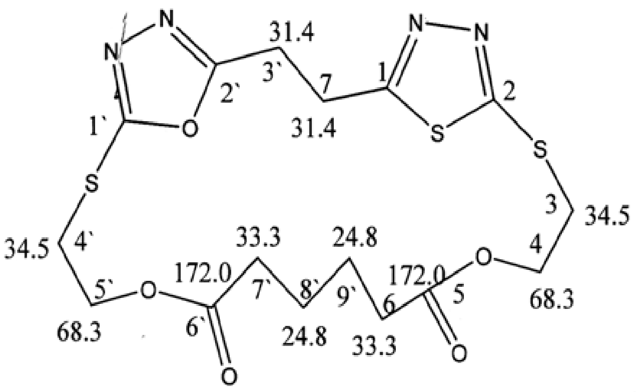


Fig (4): The 13C-N.M.R system of (L4) according to Chemoffice program

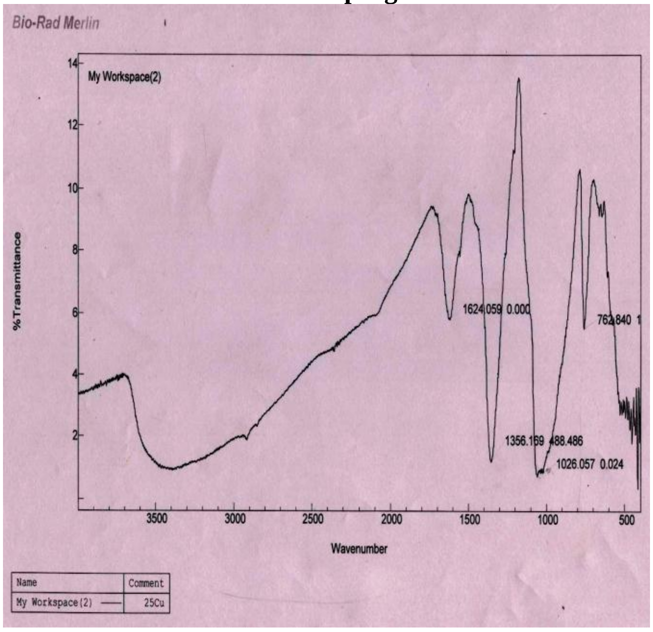


Fig (5): The IR spectrum [Cu(L3)2 Cl2] Complex

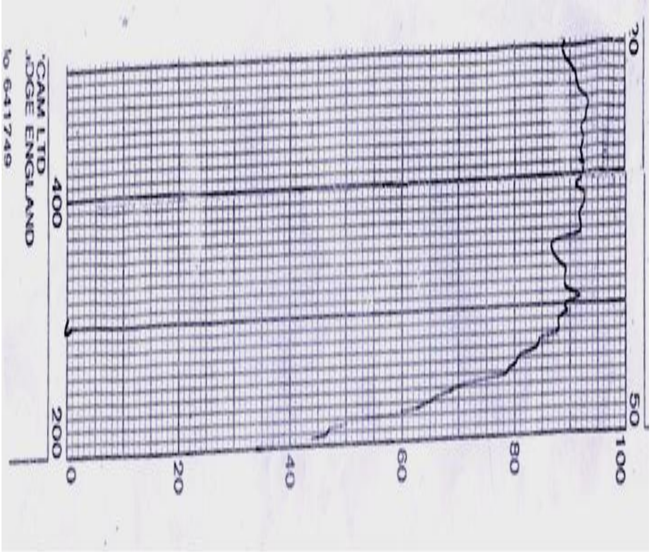


Fig (6): The IR spectrum of [Cu(L3)2 Cl2] Complex

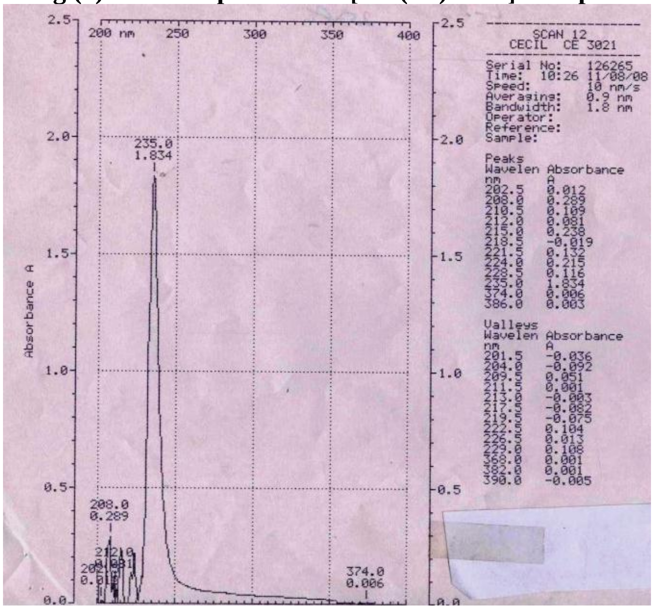
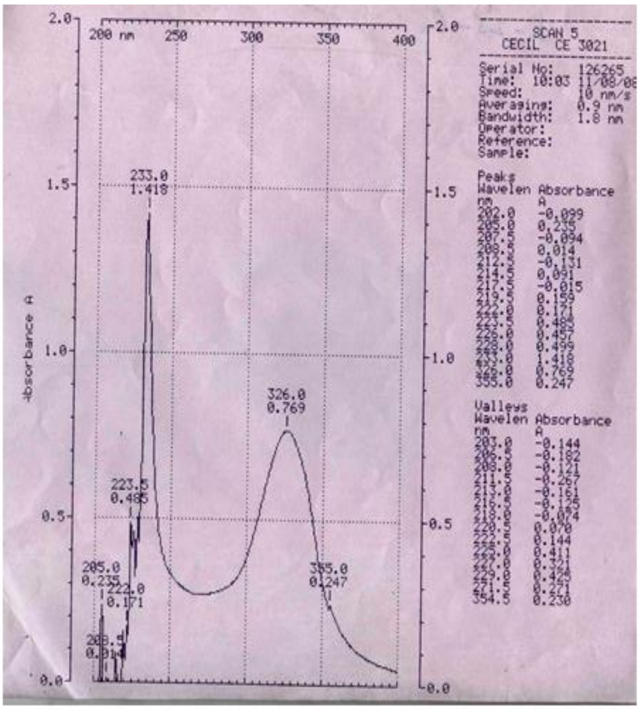


Fig (7): The UV. Spectrum of (L3)



SCAN 5
CECIL CE 3021

Serial No: 126265
Time: 10:03 11/08/08
Speed: 10 nm/s
Averaging: 0.9 nm
Bandwidth: 1.8 nm
Operator:
Reference:
Sample:

Peaks

Wavelen nm	Absorbance A
202.0	-0.099
205.0	0.235
207.5	-0.094
208.5	0.014
212.5	-0.131
214.5	0.091
217.5	-0.015
219.5	0.159
222.0	0.171
223.5	0.485
226.0	0.457
228.0	0.499
233.0	1.418
326.0	0.769
355.0	0.247

Valleys

Wavelen nm	Absorbance A
203.0	-0.144
206.5	-0.182
208.0	-0.121
211.5	-0.267
213.0	-0.161
216.5	-0.125
218.0	-0.074
220.5	0.070
222.5	0.144
225.0	0.411
227.0	0.321
229.0	0.425
271.5	0.271
354.5	0.230

Fig (8): The Uv-vis spectrum of [$Cu(L_3)_2 Cl_2$] Complex

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تحضير وتشخيص ليكاندين لاوكسادايزول ذو سلسلة جانبية مغلقة ومعقداتها مع Cu(II) and Pd (II)

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الخلاصة:

تم تحضير مشتقات ثنائي أوكسادايزول من تفاعل ١، ٢ – ثنائي (٥,٢ كلوروأثيل كبريتيد)، ٣,١، ٤–أوكسادايزول –٢– يل) أيثان(L1) و ١،٢–ثنائي –(كلوروأثيل كبريتيد)– ٤,٣,١ –أوكسادايزول –٢– يل) ميثان (L2.) مع حامض الأديبيك بوجود محلول كحولي من هايدروكسيد البوتاسيوم. وتم تصعيد مزيج التفاعل لمدة ثماني ساعات . وقد تم تشخيص الليكاندات بأستخدام اطياف IR, 1H-N.M.R و 13C-N.M.R و الكتلة. وتم استخدام الليكاندات لتحضير معقدات مع أيونات ثنائية الشحنة من ((Cu(II), Pd(II)). تم تشخيص الليكاندات والمعقدات مستخدما أطياف IR، Uv-vis والتوصيلية الكهربائية والحساسية المغناطيسية وأطياف الامتصاص الذري . وأظهرت ألنتائج بأن للمعقدات شكل ثماني السطوح.