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Preparation, Identification and screening biological activity of ligand and Metal Complexes of Co(II), Ni(II), Cu(II), Pd(II) and Pt(IV)

تحضير وتشخيص وفحص الفعالية البايلوجية لليكاند ومعقدات العناصر لكل من Co(II), Ni(II), Cu(II), Pd(II), Pt(IV)

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Abstract

A new chain of conversion metal complexes of Co(II), Ni(II), Cu(II), Pd(II),and Pt(IV), with mercaptoacetamide (H_2L) N,N'-(3,3'-dimethyl[1,1'-biphenyl]-4,4'-dil)bis(2-mercaptoacetamide) ligand, type ($N_2O_2S_2$) were prepared by condensation of (o-Tolidine) with(mercaptoacetic acid), by (2:1) metal to ligand molar ratio. The complexes were characterized by elemental analysis, magnetic susceptibility, molar conductance , IR , ^{1}H NMR and UV-Vis spectral means.

From the spectroscopic studies, Melting points, magnetic moments, physical properties and mole ratio, we suggested the octahedral Cobalt(II) , Nickel (II) , Copper(II) and Platinum(IV) Complexes with ligand and the square planar geometry suggested for Palladium (II) complex. The results submit that the metal is connective with the ligand during the carbonyl oxygen of amide (C=O), the amine group (N-H) and the (S-H) group.

الخلاصة :

سلسلة جديدة من معقدات العناصر الانتقالية لكل من (Co(II), Ni(II), Cu(II), Pd(II),Pt(IV)) مع ليكاند الأمايد (N,N'-(3,3'-dimethyl[1,1'-biphenyl]-4,4'-dil)bis(2-mercaptoacetamide) من نوع ($N_2O_2S_2$) قد تم تحضيره من تفاعل (1 مول) من اورثو توليدين مع (2 مول) من حامض الخليك الكبريتي،حيث كانت النسبة المولية نسبة الفلز : الليكاند (1:2) ، وقد تم تشخيص كل من المعقدات المحضرة والليكاند بوساطة الطرق الطيفية (تقنية الاشعة تحت الحمراء ، طيف الرنين النووي المغناطيسي، الاشعة فوق البنفسجية – المرئية) و تعيين درجة الانصهار ،الحساسية المغناطيسية ،والتوصيلية المولارية ، والتحليل الدقيق للعناصر. ومن خلال النتائج والمعطيات المتحصل عليها من التقنيات اعلاه تم الاستنتاج ان بنية ثماني السطوح لمعقد الكوبلت والنيكل والنحاس والبلاتينيوم وبنية مربع مستوي لمعقد البلاديوم .ذوات الصيغة العامة [Co_2L], [Ni_2L], [Cu_2L], [Pd_2L], [Pt_2L] النتائج المقترحه تبين ارتباط الفلز مع الليكاند من خلال مجاميع الكاربونيل والأمين والثايول.

1. Introduction

The organic ligand is one of the most developing, expanding and successful departments of science in the Preparation of coordination compounds. Synthetic organic chemistry has been investigation tremendous growth, not only in expression of carbon-hetero and carbon-carbon atom bonds but also in terms of development of new designing, catalysts, reagents, transformations and technologies [(1)].

Amide bases have played animated function in the expansion of organic chemistry preparation[(2-6)].

The compounds which include amine, aldehyde and amide class as an active group have been found to take donor characteristic and have a large variety of biological activities[(7-9)].

The compounds which include, amide group as thiourea ,urea, nicotinic acid and thiosemicarbazide have been attainment widely because of their fast coordinating inclination [(10)]. Amides play a necessary function in nature [(11)]. They are found in all living cells such as blood, skin, nerves, muscles, enzymes, antibodies and many harmones. Metal or nonmetal amide are compounds which include one or more –$CONH_2$ ligand groups or easy derivative [such as –

CONHR, -$CONR_2$, where R = methyl, phenyl, $SiMe_3$ etc.) joined to metal. Amides of potassium and sodium are the top samples of metal amides[12]. The study of Mannich reaction attracted the chemist due to their enormous range of medicinal and industrial activities [13 – 18]. It has been announce that many Mannich compounds have advantage such as anti-tumor, anti-inflammatory, anti-fungal, anti-viral, anti-bacterial, anti-convulsant, antitubercular,, antimicrobial, anti-leishmanial and anti-cancer.[19]

2. Experimental part:

1. Materials

Chemicals were supplied by Merck and BDH Chemicals Co. and used as received. [o-Tolidine, mercaptoacetic acid , Methanol, Absolute ethanol, Benzene and Ethyl acetate, hexane, dioxane, $CoCl_2.6H_2O$, $NiCl_2.6H_2O$, $CuCl_2.2H_2O$, $PdCl_2.H_2O$, $K_2PtCl_6.6H_2O$].

2. Apparatus

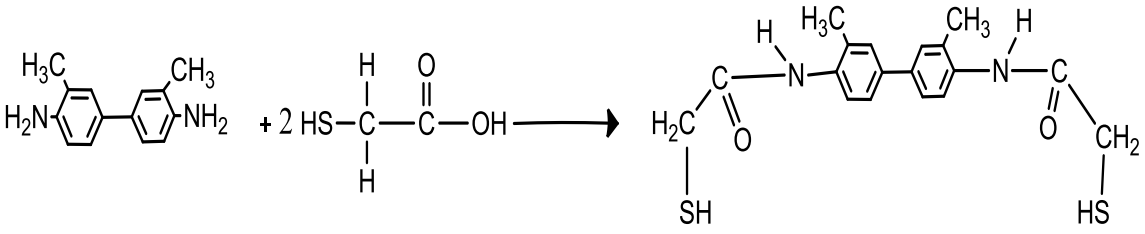
UV-Vis.(Shimadzu), Kerbala University. FT-IR spectra were recorded on FT-IR 8400s, schimadzu- spectrophotometer and using KBr discs method, in University of Kufa. ^{1}H NMR spectra were recorded on JNM - model \ Joal 400 MHZ using tetramethyl silane as internal standard and DMSO as solvent. Measurements were made at sapala organics private (India).

The Melting point of compounds produced was measured by using Gallen kamp melting point, in the University of Kerbala. The molar conductivity for complexes were measured using inluba WTW balance, in the University of kerbala. The elemental analysis (E uroEA Elemental Analyser), in the University of Kufa. the magnetic susceptibility of prepared complexes were measured by using magnetic sufcetitility balance, in the University of AL-Nahrain.

3. Synthesis Processes.

3.1. Preparation of the ligand (H_2L)

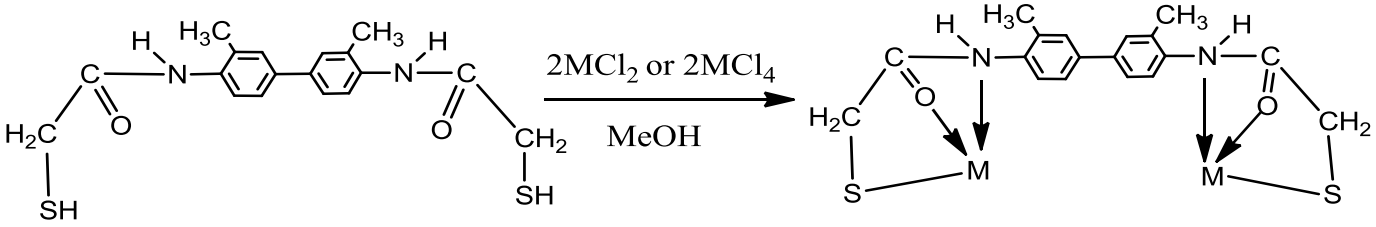
o-Tolidine (0.25 g ,0.0012 mol) was dissolved in 20 ml ethanolic solution with two drops of glacial acetic acid, then (0.22 g , 0.0023 mol) of mercapto acetic acid was added. The reaction mixture was with stirring for 1 h on a water bath at (0-5)°C. The product was then allowed to cool down to room temperature and the colored precipitate was filtered , the product was recrystallized from ethanol and followed by drying at 40 ^{o}C over 5 h.



Scheme 1- Synthesis of ligand.

3.2. Preparation of metal complexes

Co(II), Ni(II), Cu(II), Pd(II), and Pt (IV) complexes were synthesized by the reaction of (2 mol) of corresponding metal ($CoCl_2.6H_2O$, $NiCl_2.6H_2O$, $CuCl_2.2H_2O$, $PdCl_2.H_2O$, $K_2PtCl_6.6H_2O$) which were dissolved in (5-10) ml of methanol ,with a (1 mol) of the ligand was dissolved in 30 mL of methanol. The reaction mixture was refluxed and stirring for 2 h at 65°C The products were filtered ,washed with ethanol and dried at 40 ^{o}C through 6 h The physical data for the H_2L and its complexes are



listed in Table 1.

Scheme 2- Synthesis of Complexes.

M =Co^{+2} , Ni^{+2} , Cu^{+2} , Pd^{+2} , Pt^{+4}

The complexes are soluble in hot ethanol and methanol, highly soluble in DMF and DMSO , insoluble in water.

Table- 1. The physical properties and magnetic susceptibility of the ligand (H_2L) and their complexes.

Compound	Molecular formula	Molecular weight	Color	Melting point	ueff B.M	Yield %
H_2L	$C_{18}H_{20}N_2O_2S_2$	360.49	Light brown	269-272	-	85
[Co_2L]	$C_{18}H_{26}N_2O_2S_2Co_2$	483.95	Dark blue	280-283	3.70	62
[Ni_2 L]	$C_{18}H_{26}N_2O_2S_2Ni_2$	483.86	Dark green	>300	2.79	77
[Cu_2 L]	$C_{18}H_{26}N_2O_2S_2Cu_2$	483.57	Dark violet	288-291	1.67	96
[Pd_2 L]	$C_{18}H_{18}N_2O_2S_2Pd_2$	571.32	Dark brown	278-280	0.98	70
[Pt_2 L]	$C_{18}H_{26}N_2O_2S_2Pt_2$	756.65	Dark orange	>300	0.24	53

4. Results and Discussion

4.1 Infrared spectra of ligand and metal complexes:

The properties of IR absorption bands for the free ligand have been compared with those of the metal complexes in order to get information regarding the real donor atom sites of the coordinated ligand molecules. The important of IR absorption frequencies measured for the metal complexes are supply in Table (2). The sharp absorption band showed at 3250cm^{-1} in the spectrum of the free ligand is belong to the NH_2 group while the spectrum of complexes were shifted to the lower level frequency due to the coordination. The absorption band of (C=O) group in the amide observed at 1548cm^{-1} in the spectrum of ligand while the carbonyl of amide in the complexes were shifted to the lower level frequency which give good evidence that the coordination were happened. The week absorption band appeared at 2623cm^{-1} in the spectrum of the ligand is belongs to the (S-H) group but this band disappeared due to coordination . The new bands about (642 -755),(596-638) and (486-543)cm^{-1} in the spectra of the metal complexes were specific to υ(M-N), υ(M-O), υ(M-S) stretching vibrations respectively[20].

Table - 2. IR frequencies of the bands (cm^{-1}) of H_2L and their complexes

Com.	υ(N-H)	υ(S-H)	υ(C=O)	υ(C-H) aliphatic	υ(C-H) aromatic	υ(C-N)	υ(N-M)	υ(O-M)	υ(S-M)
H2L	3250	2623	1548	2904	3045	1384	-	-	-
[Co_2 L]	3115	-	1500	2668	2956	1369	675	570	540
[Ni_2 L]	3210	-	1527	2885	3028	1384	642	576	503
[Cu_2 L]	3230	-	1523	2928	3036	1327	755	638	486
[Pd_2 L]	3218	-	1536	2883	3025	1384	669	596	507
[Pt_2 L]	3111	-	1524	2868	2956	1375	671	611	543

4.2 ^{1}H NMR spectra of ligand and metal complexes:

The ^{1}H NMR spectra of the compounds were obtained in DMSO- d_6 using TMS as criterion. The chemical shift which notice for the(SH) protons in ligand (1.6 ppm) but was not notice in any of the complexes. This indicates to the disappearance of hydrogen because of the chelation between the sulfur and metal ions (S-M). The same product was assured by the IR spectra.

The spectrum of ligand display signal at (7.16 ppm) for the amide –NH(CO) while the spectrum of complexes exhibit shifting of the signals of the amide –NH(CO) protons, at (6.42 – 6.73 ppm) because of chelation[21].

In ligand and complexes, the signals of aromatic protons appeared at the domain (7.4 – 7.6 ppm), the proton peak of ($–CH_2$) at (3.3 – 3.8 ppm) and the proton peak of ($–CH_3$) at (2.3 – 2.8 ppm) they were shifted by coordination. The ^{1}H NMR spectra data of the ligand and its complexes are outlined in Table 3.

Table - 3. ^{1}H NMR spectral data of ligand (H_2L) and their complexes

Compound	SH	Aromatic .H	$–CH_2$	$–CH_3$	N-H
H_2L	1.62	7.51-7.62	3.35	2.35-2.3	7.16
[Pd_2 L]	-	7.47-7.57	3.73	2.61-2.54	6.42
[Pt_2 L]	-	7.55-7.69	3.89	2.81	6.73

4.3 Electronic spectra

The electronic spectral data of the ligand and its complexes are outlined in Table 4. The spectrum of the H_2L exhibit 2 major peak: at (375, and 421) nm. The first peak at (375nm) (26736 cm^{-1}) was assigned to the aromatic nucleus ($\pi– \pi^*$) transition. the second band in the spectrum of H_2L (421 nm) (23808cm^{-1}) was belong to($n – \pi^*$) transition in carbonyl group (C=O).

Electronic spectrum for [Co_2L] found one absorption band which is d-d transition in (457nm) (21881 cm^{-1}) where this band can be assigned to electronic transition $^{4}T_1 \rightarrow {}^{4}T_1^{p}$ in Co(II) complex which have octahedral[22]. While electronic spectrum for [Ni_2L] display two bands of absorption, the first band (430-460nm) (23255- 21739 cm^{-1}) this belongs to $^{4}T_1 \rightarrow {}^{4}T_2$, while the second band (480-500 nm) (20833 – 20000 cm^{-1}) belong to $^{4}T_1 \rightarrow {}^{4}T_1^{p}$., where it refers to the complex have octahedral shape. The spectrum of [Cu_2L] has a broad band (450 nm) (22222cm^{-1}) Because of a collection of transitions, $^{2}E \rightarrow {}^{2}T_2$. These transitions indicate distorted octahedral around Cu (II) ion[23]. The spectrum of [Pd_2L] display absorption band in (445nm) (22471cm^{-1}) belonging to $^{4}A_2 \rightarrow {}^{4}T_2$ transition and in (486nm) (20576cm^{-1}) belonging to $^{4}A_2 \rightarrow {}^{4}T_1$ transition and band in(514nm) (19455cm^{-1}) belonging to $^{4}A_2 \rightarrow {}^{4}T_1^{P}$ transition . Therefore proposed complex form a square planar[24]. Display the spectrum of Pt (IV) complex that shows two bands in (498,530) nm, (20080,18867) cm^{-1} respectively belonging to $^{4}A_2 \rightarrow {}^{4}T_1$, $^{4}A_2 \rightarrow {}^{4}T_1^{p}$ transitions therefore proposed complex form octahedral[25] .

Table - 4. Electronic spectral data of ligand (H_2L) and their complexes

Compound	λ_{max} nm	λ_{max} cm^{-1}	ε L / mol.cm	Proposed Structure
H_2L	375 421	26736 23808	13600 12500	-
[Co_2L]	457	21928	4500	OCT
[Ni_2L]	430 500	23000 20000	4800 3400	OCT
[Cu_2L]	500 626	20000 16000	4700	OCT
[Pd_2L]	445 486 535	22571 20596 19492	2900 3200 3100	Sq.p
[Pt_2L]	498 530	20100 18850	2700 650	OCT

Table - 5. Molar Conductivity Measurements in DMSO-d_6

compound	Conductivity $cohm^{-1}.cm^2.mol^{-1}$
$[Co_2 L]$	7.2
$[Ni_2 L]$	4.6
$[Cu_2 L]$	1.7
$[Pd_2 L]$	3.3
$[Pt_2 L]$	75.8

Table – 6. Micro Analytic (CHN) data.

Compound	C%		H%		N%	
	Cal.	Exp.	Cal.	Exp.	Cal.	Exp.
H_2L	59.97	59.95	5.59	5.57	7.77	7.74
$[Co_2L]$	45.39	45.36	3.81	3.77	5.88	5.85
$[Ni_2 L]$	45.43	45.40	3.81	3.79	5.89	5.86
$[Cu_2 L]$	44.52	44.50	3.74	3.70	5.77	5.75
$[Pd_2 L]$	37.84	37.81	3.18	3.16	4.90	4.87
$[Pt_2 L]$	28.88	28.85	2.42	2.40	3.74	3.70

4. Biological testing:

The biological activity studies of ligand and their metal complexes[26] against the Gram positive bacteria *S. aureus*, *B. subtilis* and Gram negative bacteria *E.coli*, *P. auroginosa*. using modifical Mueller-Hinton agar diffusion method[27].The zone of inhibition was measuring in mm , and are determined by (-), (+),(+++) according to the diameter.

The biological activity data listed in Table (7). The antibacterial activity of complexes are shows a good antibacterial activity. [Pt L] complex showed high activity compared to all the remaining synthesized compounds.

Table – 7. Antibacterial Activity of Ligand and its metal complexes

Compound	Staphylococcus aureus	Pseudomonas aereuguinosa	B. Subtiles	E. Coli
$[H_2L]$	+	-	-	+
[Co L]	+	+	-	+
[Ni L]	-	+	+	+
[Cu L]	+	-	+	+
[Pd L]	+	+	+	+
[Pt L]	+++	+++	+++	+++

The suggested geometry for the prepared complexes

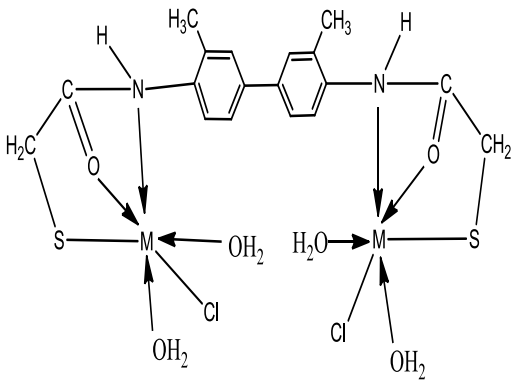


Figure 1. Structural of $[M_2L]$
M= Co^{+2} , Ni^{+2} , Cu^{+2}

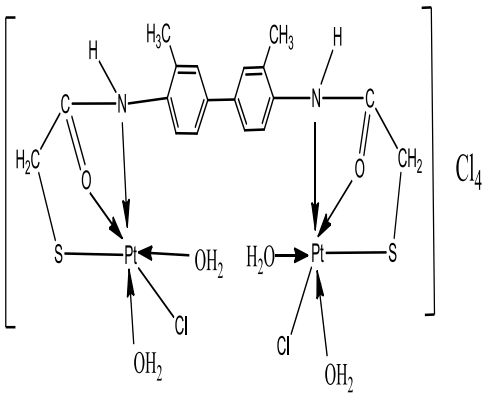


Figure 2. Structural of $[Pt_2L]\ Cl_4$

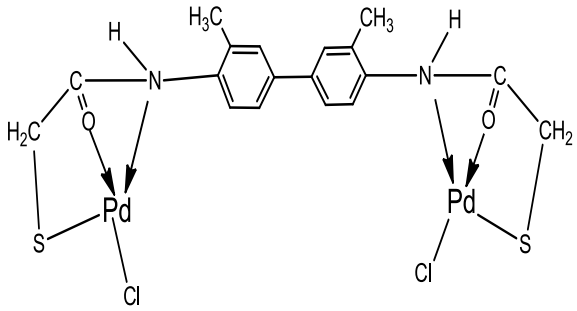


Figure 3. Structural of $[Pd_2L]$

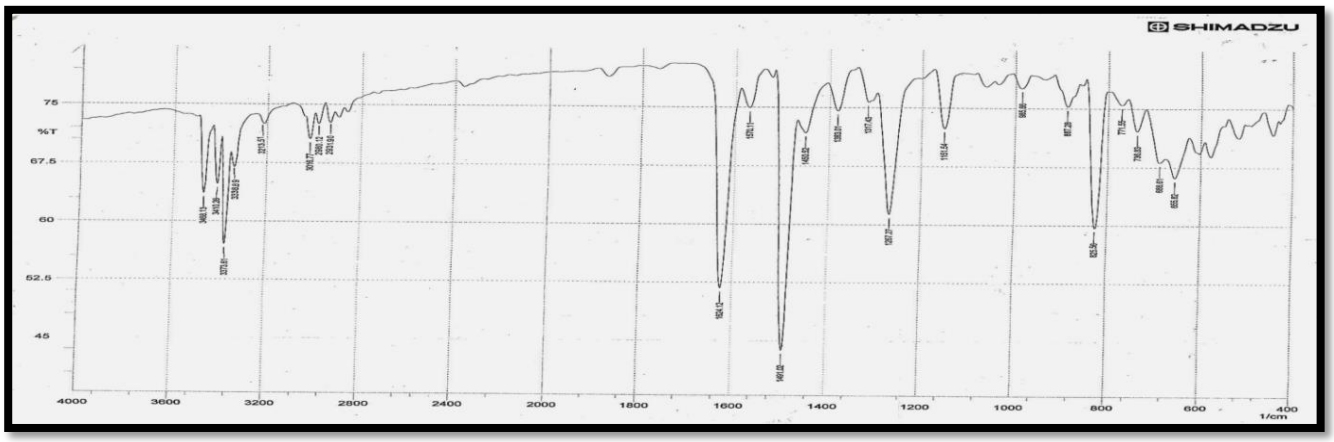


Figure 4: FT-IR spectrum of *O*- Tolidine

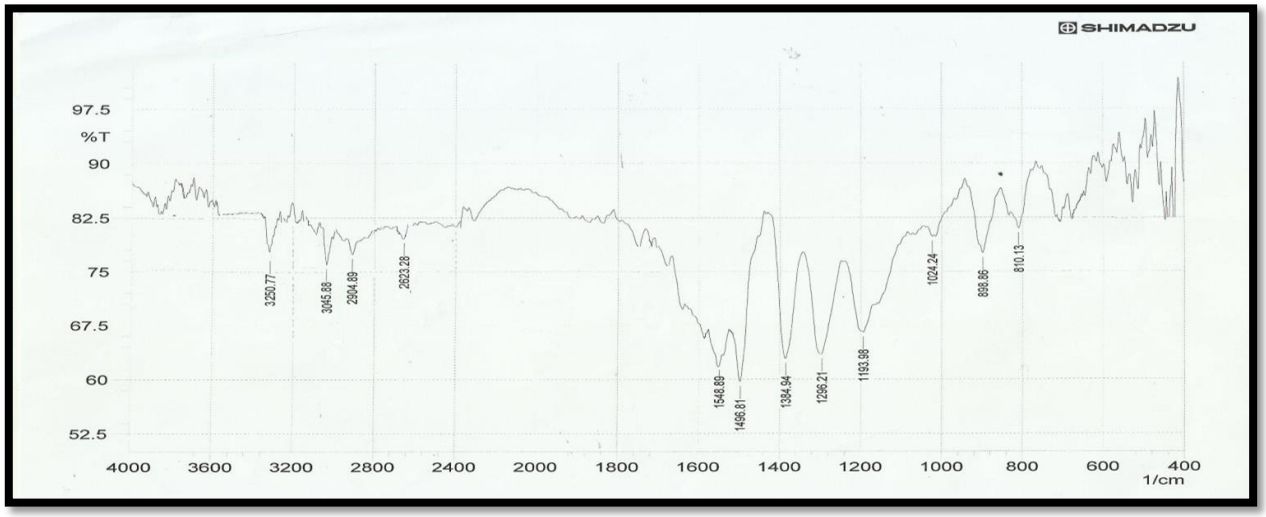


Figure 5: FT-IR spectrum of compound [H_2L]

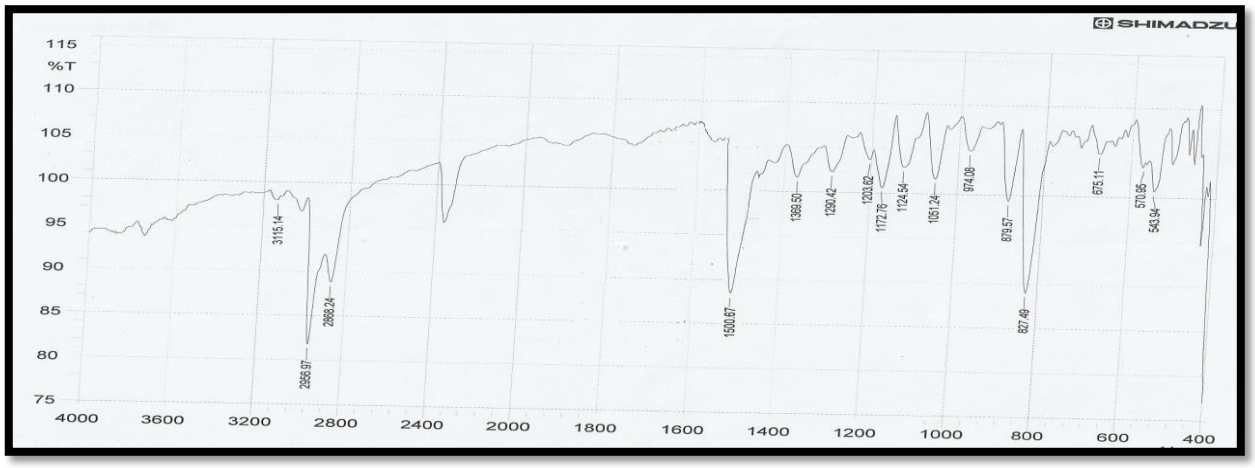


Figure 6: FT-IR spectrum of compound [Co_2L]

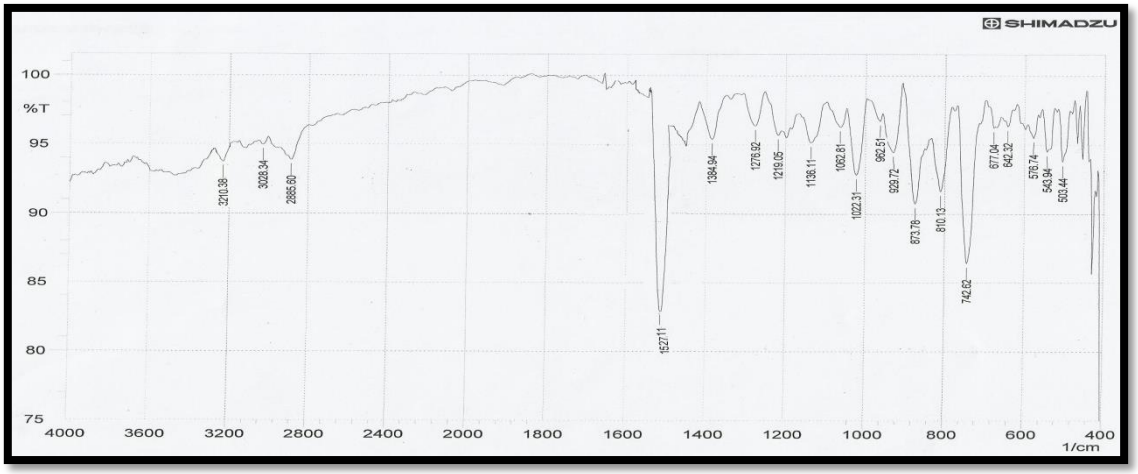


Figure 7: FT-IR spectrum of compound [Ni_2L]

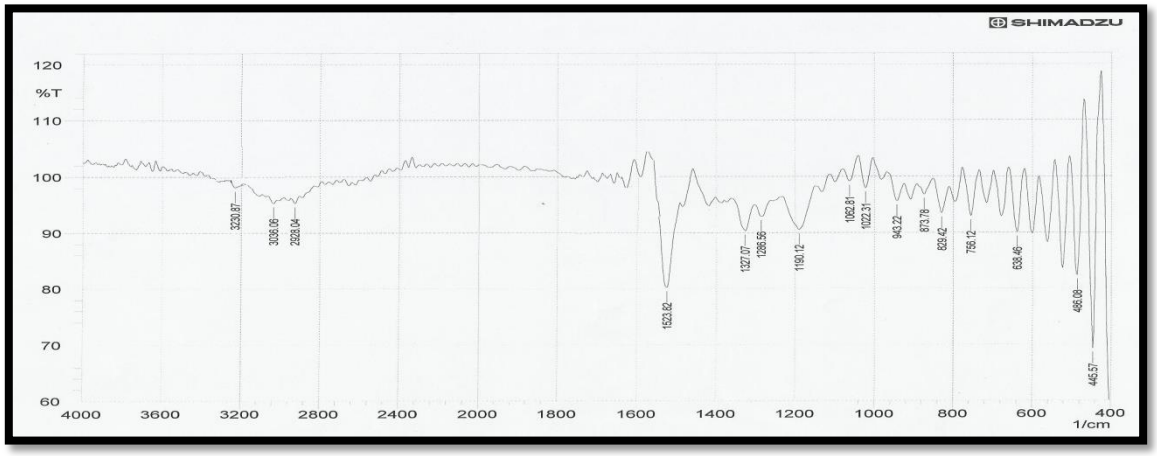


Figure 8: FT-IR spectrum of compound [Cu_2L]

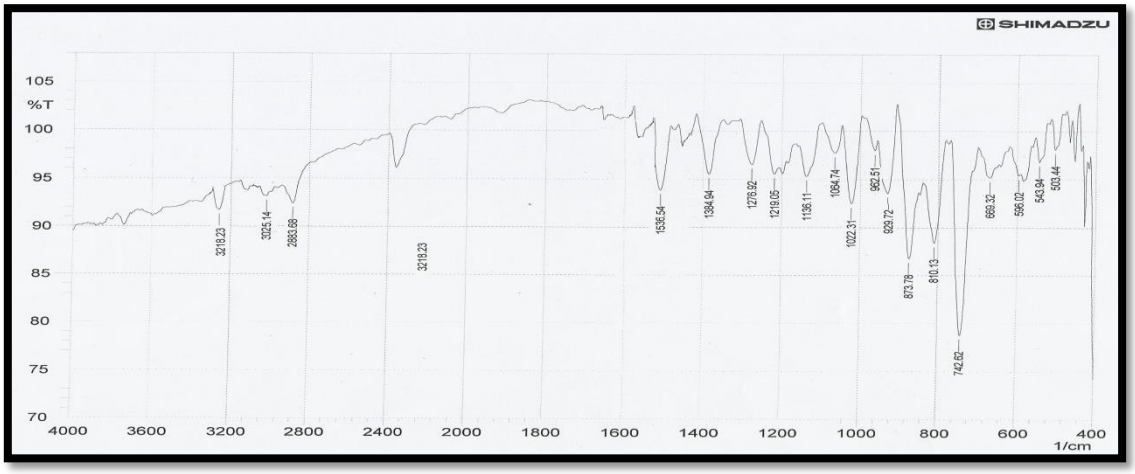


Figure 9: FT-IR spectrum of compound [Pd_2L]

Figure 10: FT-IR spectrum of compound [Pt_2L]

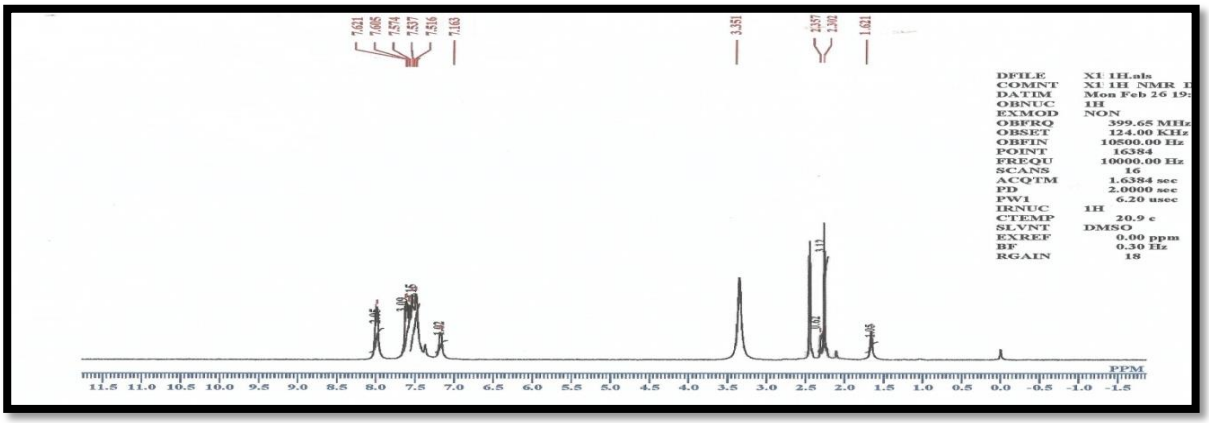


Figure 11: ^{1}H NMR spectrum of compound (H_2L)

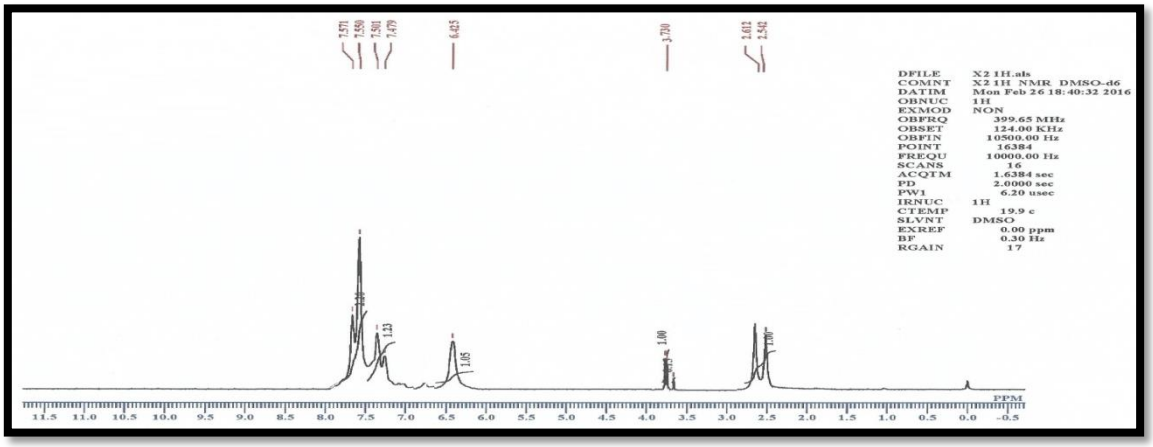


Figure 12: ^{1}H NMR spectrum of compound [Pd_2L]

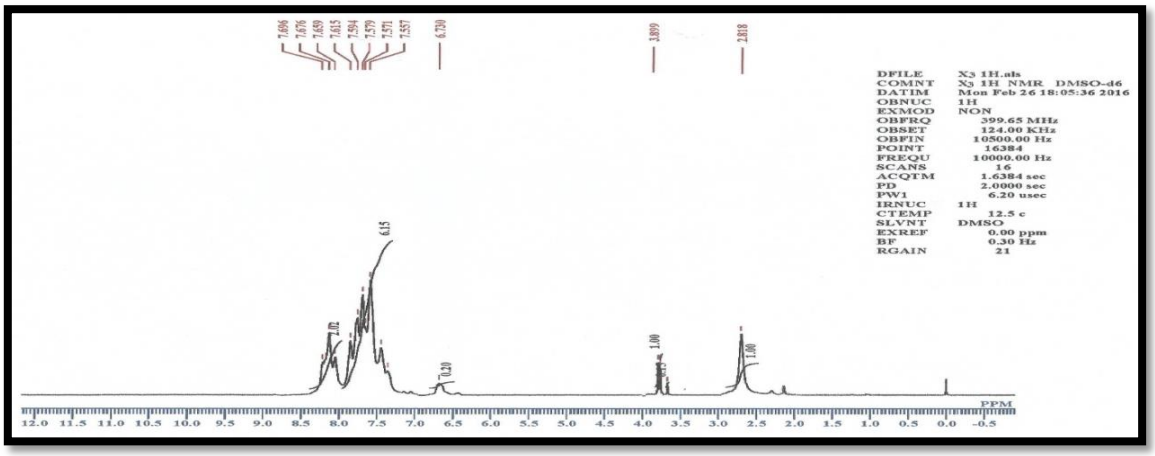


Figure 13: ^{1}H NMR spectrum of compound [Pt_2L]

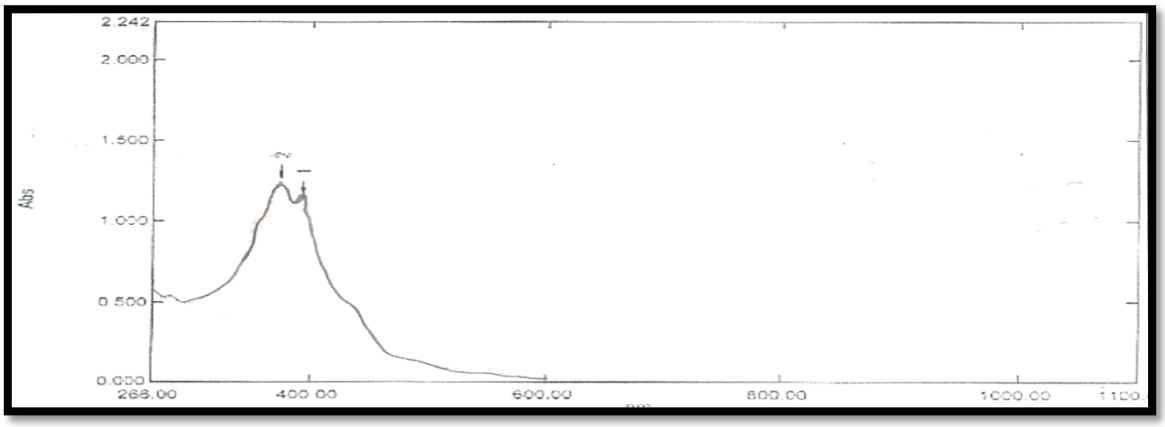


Figure 14: UV –vis spectrum of ligand (H_2L)

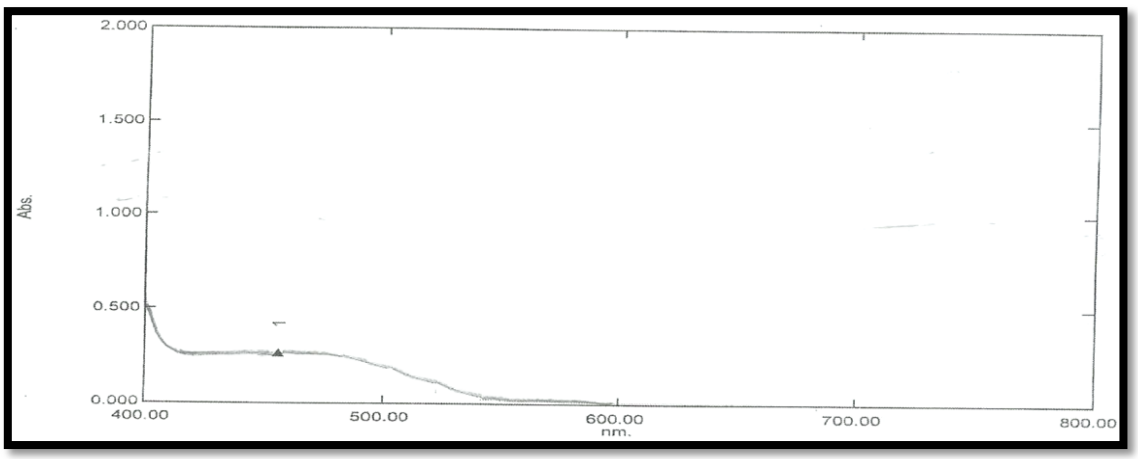


Figure15: UV –vis spectrum of Complexes[Co_2L]

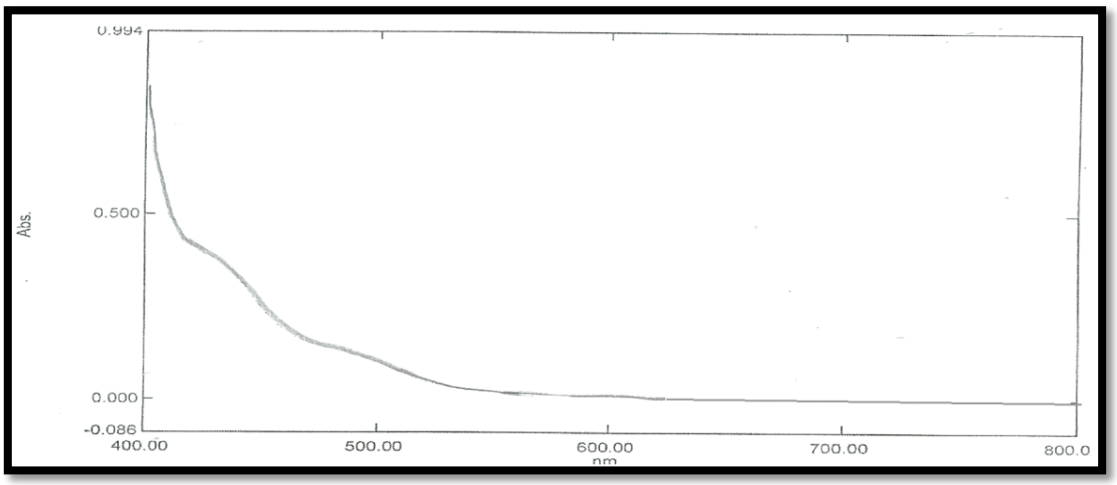


Figure16 : UV –vis spectrum of Complexes[Ni_2L]

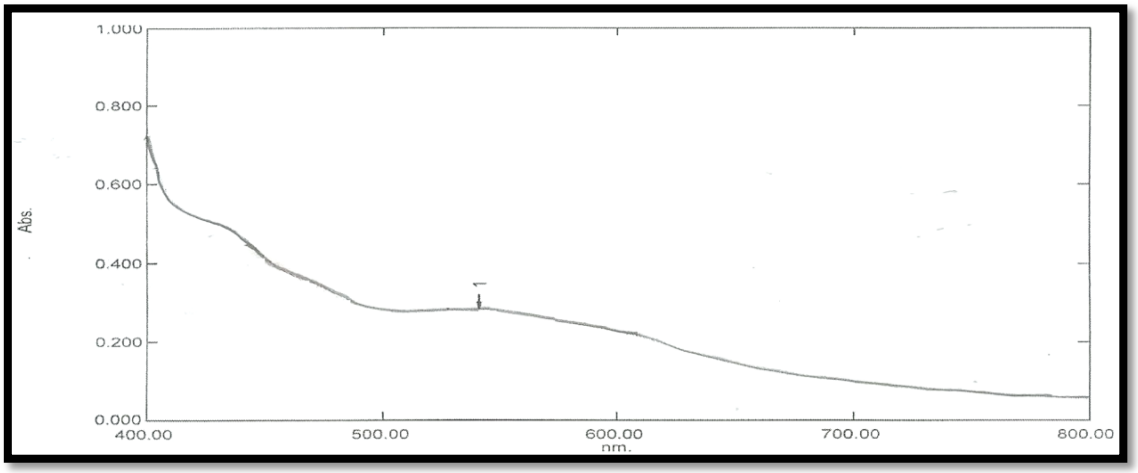


Figure17 : UV –vis spectrum of Complexes[Cu_2L]

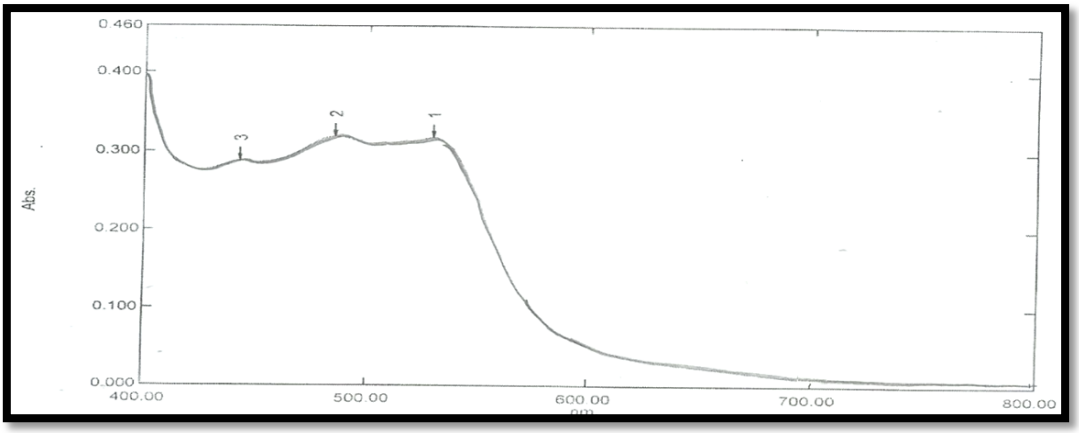


Figure18 : UV –vis spectrum of Complexes[Pd_2L]

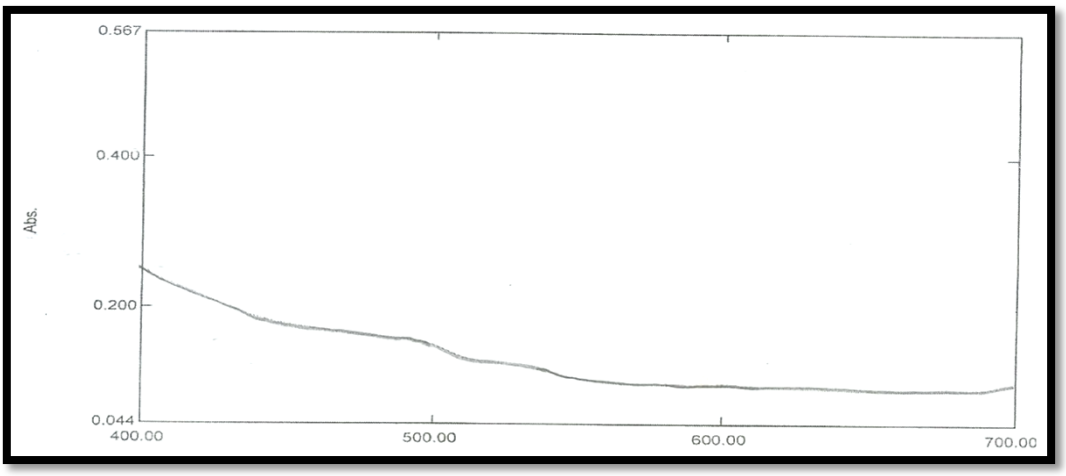


Figure19 : UV –vis spectrum of Complexes[Pt_2L]

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