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Synthesis of some bivalent Palladium (II) and Platinum (II) Complexes with Active Schiff's Base Ligand in order to Evaluate their Antibacterial Activity

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ABSTRACT

The research article represents the synthesis, characterisation and antibacterial studies some bivalent Palladium (II) and Platinum (II) complexes of active Schiff's base ligands of the type[Pd(L1H)2]Cl2 ,[Pd(L2H)2]Cl2 and [Pt(L3H)2]Cl2, [Pt(L4H)2]Cl2 derived from (2E)-1-(4-bromophynyl)-3-(4-methoxyphynyl) prop-2-en-1-one and (2E)-3-(4-methoxyphynyl)-1-(4-nitrophynyl) prop-2-en-1-one. Where L1H= hydrazinecarbothiamide of (2E)-1-(4-bromophynyl)-3-(4-methoxyphynyl) prop-2-en-1-one & L2H = hydrazinecarboxamide of (2E)-1-(4-bromophynyl)-3-(4-methoxyphynyl) prop-2-en-1-one, L3H= hydrazinecarbothiamide (2E)-3-(4-methoxyphynyl)-1-(4-nitrophynyl) prop-2-en-1-one and L4H= hydrazinecarboxamide (2E)-3-(4-methoxyphynyl)-1-(4-nitrophynyl) prop-2-en-1-one.All complexes reported here had been characterised on the basis of elemental analysis, molecular weight determinations ,by 1H NMR and FTIR spectra .Molar conductance studies supports the 1:2 nature of these complexes. On the basis of these data it reveals that Pd(II) and Pt(II)complexes were diamagnetic in nature with square planner geometry. The FTIR spectral data reveals that all the Schiff's bases (L1H, L2H, L3H & L4H) behave as a bidentate ligands and were coordinated to Pd(II) and Pt(II) metal through the sulfur and hydrogenic nitrogen atom . All the new synthesized compounds were screened for antibacterial activity against the test organism viz Escherichia coli NCIM 2641, Staphylococcus aureus MTCC 1144.

Keywords: Schiff's base, Semicarbazone, Thiosemicarbazone, Escherichia coli NCIM 2641, Staphylococcus aureus MTCC 1144.

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INTRODUCTION

The synthesis and structural investigation of some Palladium and Platinum complexes with Semicarbazone and Thiosemicarbazones based active Schiff's base ligands are of significant attention because of their pharmacological, antibacterial [1], antifungal [2], antitumor [3],antiarthritic [4] antiamebic [5] ,antiviral [6] , antimalarial properties [7] and modes of bonding and stereochemistry [8] and some have been found to possess anti-HIV activity [9]. a wide range of Schiff bases have been synthesized and their complexation behaviour were studied[10-11] because of their great flexibility, important biological and catalytic activity and wide spectrum of activities. Most of metal complexes of Semicarbazones and Thiosemicarbazones were colored and used as analytical reagents [12] for selective and sensitive determinations of metal ions. The geometry of the studied metal complexes were greatly influenced by the nature of the ligand and a variation of charge density around the coordination site and by the type of metal salts used in their preparation. The synthesized ligands and complexes were characterized and identified by using ^{1}H-NMR spectroscopy and FTIR spectroscopy, elemental analysis, molar conductivity measurements. The ligands have two nitrogen and Sulphur/ oxygen donor sites, which can effectively coordinate to a metal ion in a tetra dentate fashion. Palladium (II) and Platinum (II) complexes of L_1H= hydrazinecarbothiamide of (2E)-1-(4-bromophynyl)-3-(4-methoxyphynyl) prop-2-en-1-one, L_2H = hydrazinecarboxamide of (2E)-1-(4-bromophynyl)-3-(4-methoxyphynyl) prop-2-en-1-one, L_3H= hydrazinecarbothiamide (2E)-3-(4-methoxyphynyl)-1-(4-nitrophynyl) prop-2-en-1-one and L_4H= hydrazinecarboxamide (2E)-3-(4-methoxyphynyl)-1-(4-nitrophynyl) prop-2-en-1-one, have been prepared and screened for their antibacterial activity against the test organism viz *Escherichia coli* NCIM 2641, *Staphylococcus aureus* MTCC 1144 by paper disc diffusion technique. The antibacterial data reveals that that the complexes were superior to the free ligands .the enhanced activity of the metal complexes may be due to the lipophilic nature of these complexes arising due to the co-ordination .it was also noted that sulphur containing ligands as well as their complexes were more reactive than their oxygen counterparts. [13**]**.In the present investigation we have synthesized and characterize some new active Schiff's base complexes of Palladium and Platinum metals in order to evaluate their antibacterial activity.

EXPERIMENTAL

All chemicals used were of A.R Grade and purchased from S.D Fine and Lobachem chemicals (Mumbai) and were used further purification. This experimental part divided in to three parts,

A] Preparation of Chalcones

i] (2E)-1-(4-bromophynyl)-3-(4-methoxyphynyl) prop-2-en-1-one [C_1]:

Procedure:

In the three necked round bottom flask a mixture of 4-methoxy benzaldehyde (0.1M) and 50mL ethanol were taken and stirred for one hour. Meanwhile the solution of 20% caustic soda solution (25ml) mix with 4-bromo Acetophenone (0.1Mol) was prepared, Above prepared solution was added slowly to ethanolic solution of 4-methoxy benzaldehyde maintaining temp.15-20^{o}c, then the reaction mass was refluxed on water bath for 2-3 hrs. After the reaction reached completion (monitored by TLC), the mixture was cooled on ice salt bath. It was filtered and washed with water & Chalcone obtained was recrystallized with ethyl acetate.

ii] (2E)-3-(4-methoxyphynyl)-1-(4-nitrophynyl) prop-2-en-1-one [C_2]:

Procedure:

In the three necked round bottom flask a mixture of 4-methoxy benzaldehyde[14-17] (0.1M) and 50mL ethanol were taken and stirred for one hour. Meanwhile the solution of 20% caustic soda solution (25ml) mix with 4-nitro Acetophenone (0.1Mol) was prepared, Above prepared solution was added slowly to ethanolic solution of 4-methoxy benzaldehyde maintaining temp.15-20^{o}c, then the reaction mass was refluxed on water bath for 2-3 hrs. After the reaction reached completion (monitored by TLC), the mixture was cooled on ice salt bath. It was filtered and washed with water & Chalcone obtained was recrystallized with ethyl acetate.

B] Preparation of Ligands:

The ligands of hydrazine-carbothiamide (L_1H) of (2E)-1-(4-bromophynyl)-3-(4-methoxy)prop-2-en-1-one, hydrazine-carboxamide(L_2H) of (2^E) - 1 -(4-bromophynyl) – 3 - (4-methoxy) prop – 2 – en - 1- one , hydrazine-carbothiamide (L_3H) of (2E)-3-(4-methoxyphynyl)-1-(4-nitrophynyl)prop-2-en-1-one and hydrazine-carboxamide (L_4H) of (2E)-3-(4-methoxyphynyl)-1-(4-nitrophynyl)prop-2-en-1-one are prepared as

i) Preparation of hydrazine-carbothiamide (L_1H) of (2E)-1-(4-bromophynyl)-3-(4-methoxy) prop-2-en-1-one

The chalcone (2E)-1-(4-bromophynyl)-3-(4-methoxy)prop-2-en-1-one[18-32](0.01mol)was added to 25 ml of THF & thiosemicarbazide (0.012mol) was added along with sodium acetate (5gm) reaction mixture was then refluxed on water bath for 2-3 hrs. After the reaction reached completion (monitored by TLC); the mixture was cooled on ice-salt mixture, it was then filtered and recrystallized with alcohol.

ii) Preparation of hydrazine-carboxamide (L_2H) of (2E)-1-(4-bromophynyl)-3-(4-methoxy) prop-2-en-1-one

The mixture of chalcone (2E)-1-(4-bromophynyl)-3-(4-methoxy) prop-2-en-1-one (0.01mol) & semicarbazide hydrochloride (0.012mol) was added to 50ml THF. To that sodium acetate (5gm) was added. The reaction mixture was refluxed on water bath for 2-3 hrs. After completion of reaction (monitored by TLC); the reaction mixture was cooled, filtered & the product obtained was recrystallized by alcohol. The crystalized powder was further subjected to Silica gel column chromatography (2% EtoAc- Hexane) to get purified product.

iii) Preparation of hydrazine-carbothiamide (L_3H) of (2E)-3-(4-methoxyphynyl)-1-(4-nitrophynyl) prop-2-en-1-one

The chalcone (2E)-3-(4-methoxyphynyl)-1-(4-nitrophynyl)prop-2-en-1-one (0.01mol) was added to 25 ml of THF & thiosemicarbazide (0.012mol) was added along with sodium acetate (5gm) reaction mixture was then refluxed on water bath for 2-3 hrs. After the reaction reached completion (monitored by TLC); the mixture was cooled on ice-salt mixture, it was than filtered and recrystallized with alcohol.

iv)Preparation of hydrazine-carboxamide (L_4H) of (2E)-3-(4-methoxyphynyl)-1-(4-nitrophenyl) prop-2-en-1-one

The mixture of chalcone (2E)-3-(4-methoxyphynyl)-1-(4-nitrophynyl) prop-2-en-1-one (0.01mol) & semicarbazide hydrochloride (0.012mol) was added to 50ml THF. To that sodium acetate (5gm) was added. The reaction mixture was refluxed on water bath for 2-3 hrs. After completion of reaction (monitored by TLC); the reaction mixture was cooled, filtered & the product obtained was recrystallized by alcohol. The crystalized powder was further subjected to Silicagel column chromatograpphy (2% EtoAc- Hexane) to get purified product.

v) Preparation of $[Pd(L_1H)_2]Cl_2$ complexes:

$PdCl_2$ (0.001mol.) was added to an ethanolic [33-35] solution of ligands[**L_1H**](0.002mol.). The reaction mixture was than heated under reflux for about 6 hrs in presence of few drops of concentrated HCl. The reaction mixture was than cooled and filtered. The crystal obtained were washed several times with ice cold alcohol and dried in vacuum. Similarly the $[Pd(L_2H)_2]Cl_2$, $[Pd(L_3H)_2]Cl_2$,and $[Pd(L_4H)_2]Cl_2$complexes are prepared as discussed the above procedure.

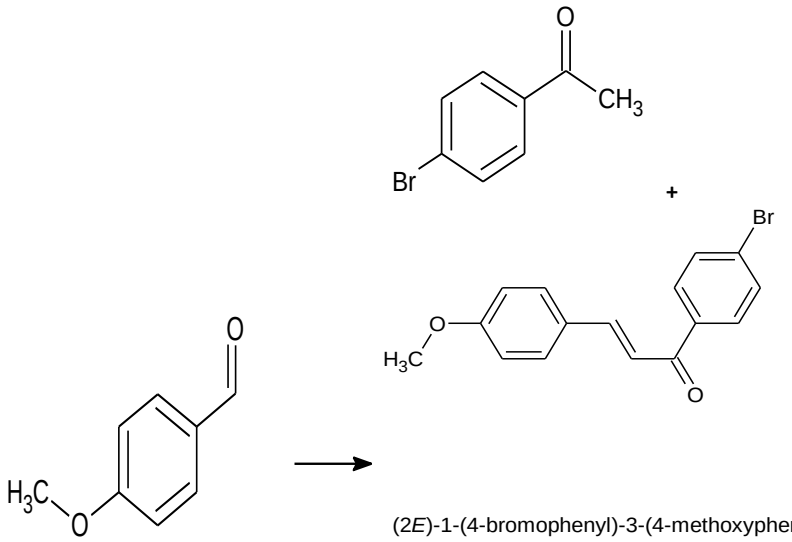
vi) Preparation of $[Pt(L_1H)_2]Cl_2$ complexes:

Procedure:

$PtCl_2$ (0.001mol.) was added to an ethanolic solution of ligand [**L_1H**] (0.002mol.). The reaction mixture was than heated under reflux for about 6 hrs in presence of few drops of concentrated HCl. The reaction mixture was than cooled and filtered. The crystal obtained were washed several times with ice cold alcohol and dried in vacuum. Similarly the $[Pt(L_2H)_2]Cl_2$, $[Pt(L_3H)_2]Cl_2$,and $[Pt(L_4H)_2]Cl_2$complexes are prepared as discussed the above procedure.

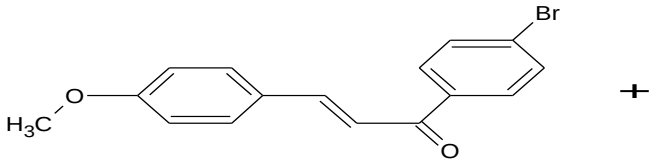
ISSN: 0975-8585

Reaction Scheme:

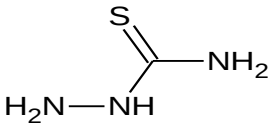


(2*E*)-1-(4-bromophenyl)-3-(4-methoxyphenyl)prop-2-en-1-one

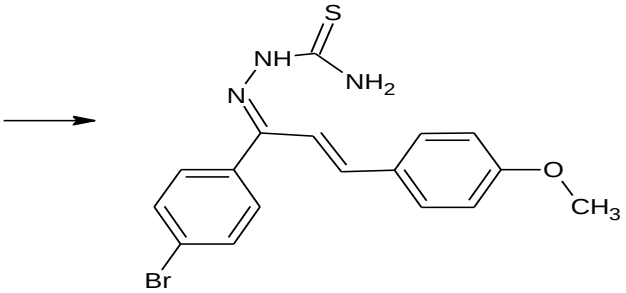
Schme-1 Preparation of chalcones.



(2*E*)-1-(4-bromophenyl)-3-(4-methoxyphenyl)prop-2-en-1-one



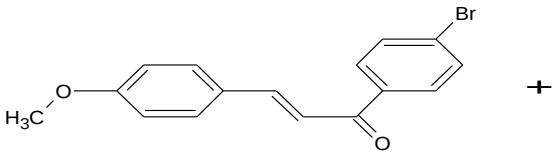
hydrazinecarbothioamide



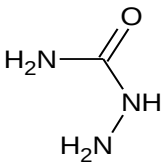
(2*E*)-2-[(2*E*)-1-(4-bromophenyl)-3-(4-methoxyphenyl)prop-2-en-1-ylidene]hydrazinecarbothioamide

Schme-2 Preparation of Ligands (L_1H).

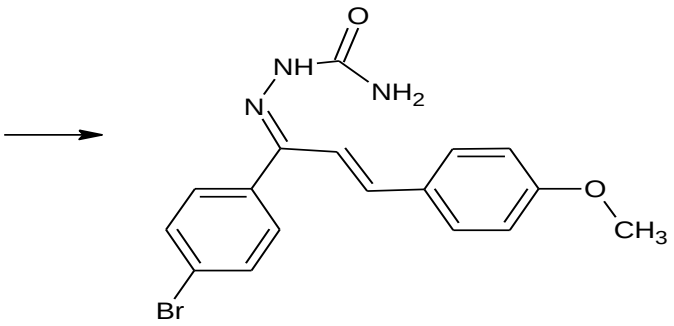
ISSN: 0975-8585



(2*E*)-1-(4-bromophenyl)-3-(4-methoxyphenyl)prop-2-en-1-one

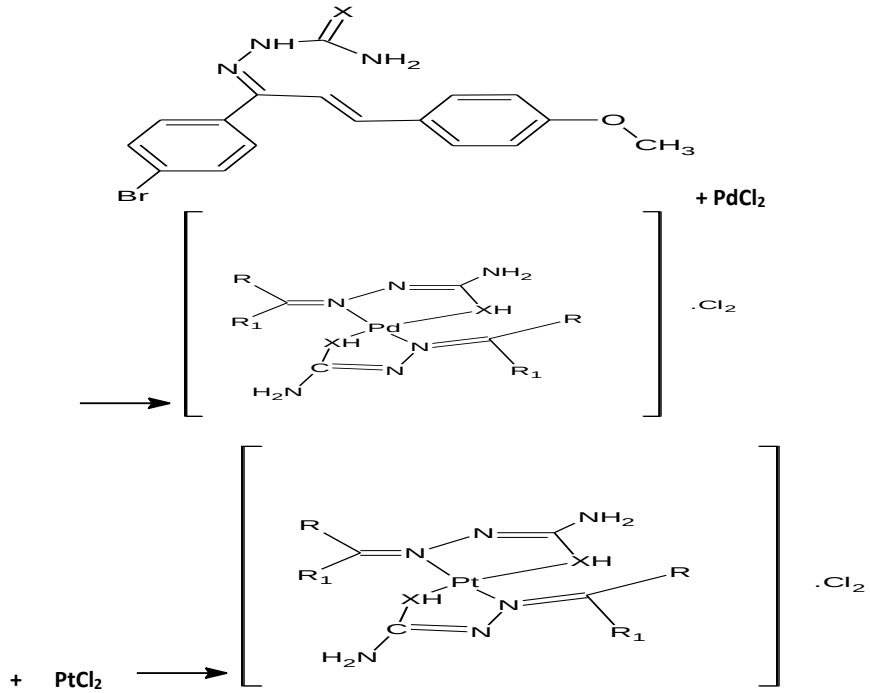


hydrazinecarboxamide



(2*E*)-2-[(2*E*)-1-(4-bromophenyl)-3-(4-methoxyphenyl)prop-2-en-1-ylidene]hydrazinecarboxamide

Schme-3 Preparation of Ligands (L_2H).



Schme-4 Preparation of $[Pd(L_1H)_2]Cl_2$ & $[Pd(L_2H)_2]Cl_2$ complexes

Product Code.

	X=S	X=O
Where R = C_6H_4Br	$Pd(L_1H)_2Cl_2$	$Pt(L_1H)_2Cl_2$

$R_1 = C_{10}H_9O$

ISSN: 0975-8585

RESULT AND DISCUSSION

The ^{1}H NMR spectra were recorded on Hitachi PerkinElmer spectrophotometer in $CDCl_3$ Using TMS as internal standard. FTIR spectra (in 4000–450 cm^{-1}range) of Ligands as well as complexes were recorded in KBr pellets (2 mg / 200 mg KBr) using a FTIR PerkinElmer 1750 spectrophotometer in department of chemistry ,University of Mumbai, Mumbai. Molecular weights were determined by the Rast Camphor method. Molar conductivities Measurements of the complexes were made with a Equiptronics Model-305.Nitrogen was determined by the Kjeldahls method and sulphur was estimated by the messengers method .Pd and Pt were estimated gravimetrically. The molar conductance values of 10^{-3}M solutions of $[M(LH)_2]Cl_2$ type complexes lie in the rage of 186-170 Sm^2mol^{-1}in dry DMF indicating 1:2 electrolytes nature .the analytical data of the ligands and complexes are given in Table-3.

i) ^{1}H NMR Spectra of $[Pd(L_1H)_2]Cl_2$ complex (400 MHz, $CDCl_3$) δ_{ppm} :-

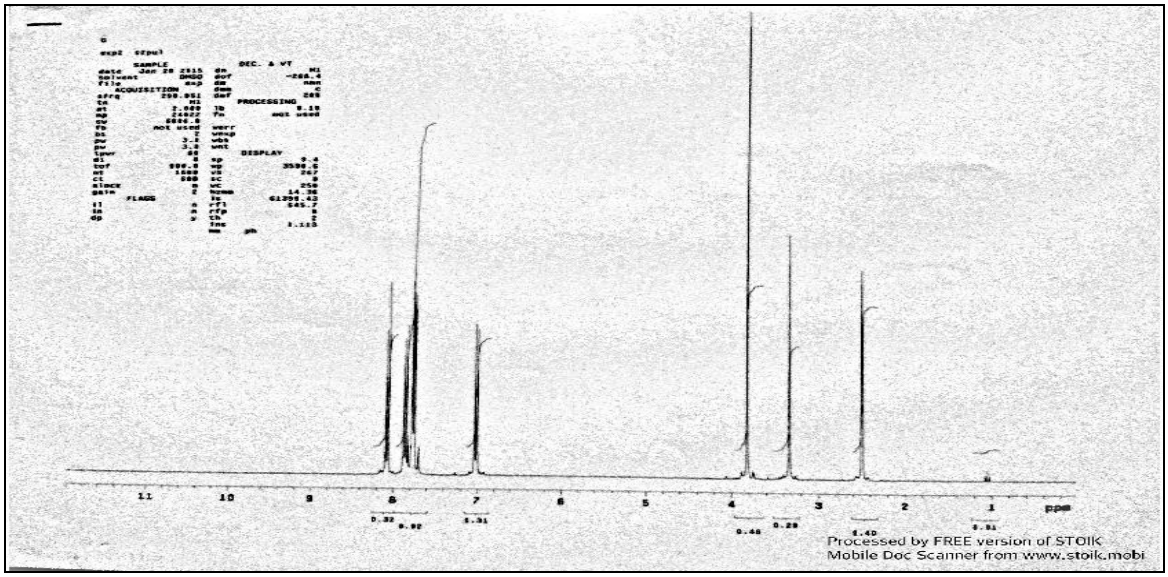


Figure: 1 NMR spectrum of $[Pd(L_1H)_2]Cl_2$ complex.

^{1}H NMR (in 400 MHz, $CDCl_3$) δ_{ppm}:-

(-0CH_3), 3H, Singlet at δ3.8, (- NH_2) 2H, broad, singlet at δ3.35, two doublet of olefinic protons (>C=C-H) at δ7.0-8.1, Aromatic ring Proton, 8H, Multiplate at δ7.7-7.9

In the ^{1}H NMR spectrum of complex the most common NMR multiplets for Aromatic rings protons are found to be resonating around δ 7.7-δ 7.9 whereas the broad singlet for –NH_2 group protons appeared around δ3.35. a sharp two doublet peak for olefinic protons (>C=C-H) group the complexes are observed in the range of δ7.0- δ8.1. a singlet for -OCH_3 appears at δ3.8. The distinguishing singlet peak around δ9.3 (>C=N-group) for azomethine protons observed in ligands was completely disappears in the complexes due to co-ordination thorough >C=N-group indicates the formation of Palladium complex. The ^{1}H NMR spectrums of $[Pd(L_2H)_2]Cl_2$, $[Pd(L_3H)_2]Cl_2$, $[Pd(L_4H)_2]Cl_2$ and $[Pt(L_2H)_2]Cl_2$, $[Pt(L_3H)_2]Cl_2$, $[Pt(L_4H)_2]Cl_2$ complexes were reported in Table -1.

ISSN: 0975-8585

Table-1: ^{1}HNMR Ligands & complexes (400 MHz, $CDCl_3$) δ_{ppm} :-

			^{1}HNMR peaks				
No	Ligands & complexes	$-NH_2$ δ_{ppm}	>NH δ_{ppm}	Olefinic Proton 1H doublet δ_{ppm}	Olefinic Proton 1H doublet δ_{ppm}	$-OCH_3$ Proton	Aromatic ring 8H Proton δ_{ppm}
1	(L_1H)	3.4	9.7	7.4	8.2	3.8	7.6-7.9
2	(L_2H)	3.3	9.4	7.3	8.1	3.75	7.5-7.7
3	(L_3H)	3.38	9.8	7.4	8.0	3.8	7.6-8.2
4	(L_4H)	3.6	9.3	7.3	8.1	3.7	7.7-8.2
5	$Pd(L_1H)_2Cl_2$	3.35	Absent	7.6	7.0-8.1	3.8	7.7-7.9
6	$Pd(L_2H)_2Cl_2$	3.4	11.3	6.8	7.8	3.8	7.1-8.1
7	$Pd(L_3H)_2Cl_2$	3.4	11.8	7.4	8.2	3.7	7.6-8.0
8	$Pd(L_4H)_2Cl_2$	3.4	Absent	7.7	8.0	3.7	7.8-8.2
9	$Pt(L_1H)_2Cl_2$	3.4	Absent	6.9	8.1	3.8	7.7-7.9
10	$Pt(L_2H)_2Cl_2$	3.4	Absent	6.8	7.8	3.7	7.1-7.6
11	$Pt(L_3H)_2Cl_2$	3.5	Absent	7.4	8.1	3.8	7.8-8.2
12	$Pt(L_4H)_2Cl_2$	3.4	Absent	7.5	8.2	3.75	7.6-8.0

ii) FTIR Spectra of $[Pd(L_1H)_2]Cl_2$ complex

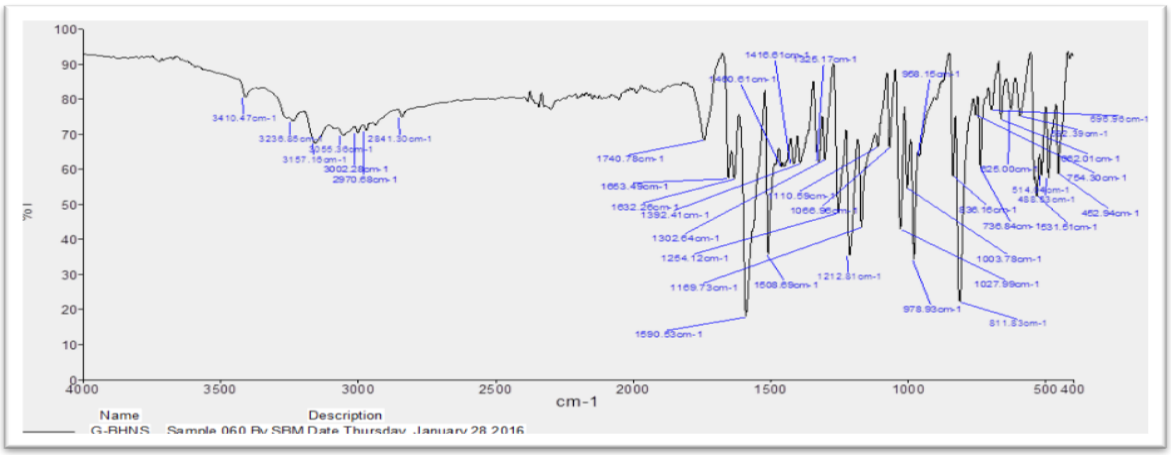


Figure: 2 FTIR spectrum of $[Pd(L_1H)_2]Cl_2$ complex.

IR (KBr) cm^{-1}:-3410-3236(>NH, NH_2), 2970 (CH), 811 (>C=S), 1590 (Aromatic Stretching), ν(C=N) groups 1509 cm^{-1}

On complexation the bands corresponding to ν(C=N) and ν(C = S) (in case of thiosemicarbazone) are shifted towards lower side1509 cm^{-1} and 811 cm^{-1} (ca.20-30 cm^{-1}). This suggest that the ligand acts as a bidentate chelating agent coordinating through nitrogen of ν(C=N) group and sulphur of ν(C = S) group. The FTIR spectrums of $[Pd(L_2H)_2]Cl_2$, $[Pd(L_3H)_2]Cl_2$, $[Pd(L_4H)_2]Cl_2$ and $[Pt(L_2H)_2]Cl_2$, $[Pt(L_3H)_2]Cl_2$, $[Pt(L_4H)_2]Cl_2$ complexes were reported in Table -2.

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Table-2: FTIR Spectrum of Ligands & complexes (in KBr)

S No	Ligands & complexes	-NH2, >NH Stretching frequency in cm-1	(>C=S) Stretching frequency in cm-1	(>C=O) Stretching frequency in cm-1	(>C=N) Stretching frequency in cm-1	Aromatic ring Stretching frequency in cm-1
1	(L1H)	3400-3250	810	----	1586	1603
2	(L2H)	3427-3279	----	1655	1580	1591
3	(L3H)	3330-3185	811	----	1583	1595
4	(L4H)	3300-3195	----	1655	1580	1603
5	Pd(L1H)2Cl2	3227-3161	809	----	1558	1600
6	Pd(L2H)2Cl2	3327-3179	-----	1655	1562	1608
7	Pd(L3H)2Cl2	3250-3100	813	-----	1555	1606
8	Pd(L4H)2Cl2	3300-3190	-----	1650	1561	1608
9	Pt(L1H)2Cl2	3270-3179	816	----	1565	1591
10	Pt(L2H)2Cl2	3327-3179	----	1655	1560	1603
11	Pt(L3H)2Cl2	3280-3170	813	---	1515	1601
12	Pt(L4H)2Cl2	3300-3150	----	1648	1513	1604

iii) ANTIBACTERIAL ACTIVITY: All the new synthesized compounds were screened for antibacterial activity [39-40] against four of the test organism viz *Escherichia coli NCIM 2641and Staphylococcus aureus* MTCC 1144.For this screening plate diffusion assay method was used [Spooner and Skyes 1972.

Antimicrobial activity by Paper Disc diffusion method:-

Disc assay method /Paper disc method [Spooner and Skyes 1972**]**

Discs were prepared by punching Whatmann filter paper no 1 at diameter of 6 mm and sterilized by autoclaving in an empty petri dish. 10 mg/ml stock solution of sample were prepared in dimethyl sulfoxide [DMSO] 24 hours old grown culture of test organism of optical density of 0.1 at 530 nm approx. cell density of $1X10^8$ CFU/ml. were surface spread on sterile and dried Mueller Hinton Agar medium. Plates were kept for adsorption of culture. 50µL of the crude extract in different solvents was added to each sterile disc .These discs, along with the positive control antibiotic discs (streptomycin, 10µg/disc and Penicillin 10 U/ml.) and negative control discs of solvent were placed on surface of plates and kept at 4˚C for 15 minutes to facilitate maximum diffusion.After plates were kept in an incubator 37˚C for 24 hrs to allow the growth of the organisms. The antibacterial activities of the test agents were determined by measuring the diameter of the zone of inhibition in millimetre**.** Results as per Table No.1shows average of diameter of zone of inhibition of triplicate set.Test culture used for test are pathogenic and normal flora, one is of Gram positive and another is of Gram negative group. The antibacterial data reveals that that the complexes were superior to the free ligands .the enhanced activity of the metal complexes may be ascribed to the lipophilic nature of these complexes arising due to the chelation [41-42].it was also noted that sulphur containing ligands as well as their complexes were more reactive than their oxygen counterparts. The Antibacterial data for the free ligands and their corresponding complexes were reported in Table -4

Table-3: Analytical and Physical Data for L_1H, L_2H, L_3H and L_4H and their complexes.

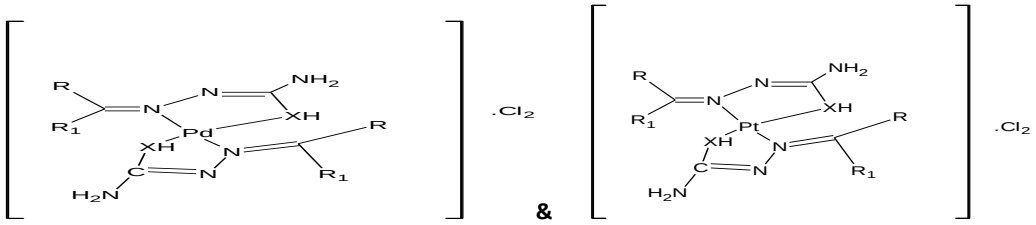
Sr. No	Free Ligands & complexes	Yield (%)	Melting Point (0C)	Molar Conductance Sm2mol-1	Found (calculated) (%)					
					M	C	H	N	S	Molecular weight Found(Calcd)
	(L1H)	58	250-255	--	--	63.38 (64.26)	4.25 (4.59)	16.77 (16.55)	9.62 (9.53)	378 (389)
	(L2H)	55	242-247	--	---	67.14 (67.78)	5.25 (5.15)	17.43 (17.35)	--	369 (373)

ISSN: 0975-8585

	(L3H)	54	232-235	--	---	63.58 (64.76)	4.38 (4.69)	16.97 (16.85)	9.22 (9.48)	344 (356)
	(L4H)	51	228-232	--	---	67.15 (67.68)	5.18 (5.42)	17.23 (17.49)	--	337 (340)
	Pd(L1H)2Cl2	40	285-288	185.2	12.31 (12.77)	50.24 (50.67)	3.26 (3.93)	13.18 (13.35)	7.06 (7.44)	867 (883)
	Pd(L2H)2Cl2	38	280-285	173.28	12.05 (12.45)	52.49 (52.93)	3.66 (3.97)	13.22 (13.83)	---	828 (843)
	Pd(L3H)2Cl2	41	275-278	170.82	12.13 (12.85)	50.34 (50.97)	3.16 (3.93)	13.08 (13.45)	7.58 (7.29)	809 (817)
	Pd(L4H)2Cl2	35	270-273	178.82	12.14 (12.51)	52.49 (52.73)	3.56 (3.87)	13.12 (13.63)	---	769 (785)
	Pt(L1H)2Cl2	41	285-290	180.23	20.15 (20.73)	46.28 (46.20)	3.69 (3.88)	11.56 (11.98)	6.54 (6.78)	778 (793)
0	Pt(L2H)2Cl2	38	290-295	189.83	21.57 (21.25)	48.20 (50.68)	4.44 (3.77)	12.37 (12.58)	--	738 (753)
1	Pt(L3H)2Cl2	39	280-284	185.73	21.38 (21.67)	46.58 (46.40)	3.58 (3.97)	11.26 (11.88)	6.24 (6.88)	714 (727)
2	Pt(L4H)2Cl2	41	275-281	186.88	21.43 (21.58)	48.12 (50.76)	4.52 (3.73)	12.45 (12.78)	--	692 (703)

Table- 4 Antibacterial activities of Ligands and their complexes

S.N.	**Sample**	**Test Culture**	
		Escherichia coli NCIM 2641	*Staphylococcus aureus* MTCC 1144
		Diameter of zone of inhibition [mm]	
1	L_1H	Nil	8
2	L_2H	Nil	8.5
3	L_3H	Nil	11.5
4	L_4H	Nil	6.5
5	$[Pd(L_1H)_2]Cl_2$	7.5	Nil
6	$[Pd(L_2H)_2]Cl_2$	Nil	18.5
7	$[Pd(L_3H)_2]Cl_2$	Nil	16.5
8	$[Pd(L_4H)_2]Cl_2$	12	7.5
9	$[Pt(L_1H)_2]Cl_2$	7	7.5
10	$[Pt(L_2H)_2]Cl_2$	Nil	9
11	$[Pt(L_3H)_2]Cl_2$	7	Nil
12	$[Pt(L_4H)_2]Cl_2$	Nil	14
Solvent control	**DMSO**	Nil	Nil
Std. Antibiotic	**Streptomycin [10ug/disc]**	15	14
	Penicillin [10 U/disc]	16	18



$[Pd(LH)_2]Cl_2$ & $[Pt(LH)_2]$ complexes

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