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Synthesis and Spectroscopic study of Pd(II)- Salicylaldoxime complexes with amine ligands

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ABSTRACT

New Pd(II) complexes of Salicylaldoxime (HSaly) with amine ligands {amine = Bipy, Phen and en} were synthesized and characterized by using CHN analysis, FT-IR spectra, molar conductivity and ^{1}H NMR spectra. The HSaly ligand was coordinated with the Pd(II) ion as bidentate chelating ligand in complex $[Pd(Saly)_2]$, through oxygen atom of hydroxylate group and nitrogen atom of oxime group, whereas, bonded as monodentate ligand in complexes $[Pd(Saly)_2(amine)]$ **(2-4)**, via the oxygen atom of hydroxylate group, and the amines were coordinated as bidentate chelate via N atom to give a square planner around the Palladium (II).

1. Introduction

Salicylaldoxime and its derivatives have been the focus of comprehensive research in coordination chemistry. Because of their richly painted complexes with a wide structural variety developed with the majority of transition metals [1-30]. Metal complexes of oxime ligands have piqued the attention of researchers because they regulate a wide variety of medical, manufacturing, and environmental areas, including the removal of metal ions from wastewater [3,5,10,13,14,19,23,26,29], as well as the use of oximes in the gravimetric determination of such metal ions [31]. Salicylaldoxime can coordinate to metals in a variety of ways [1-30](**Chart 1**) as:.

(1) Monodentate style via O atom of hydroxylate group (i).

(2) Bidentate chelate style via the oxygen atom of hydroxylate group and nitrogen of oxime set (ii).

(3) bridging mode through the one or two oxygen atoms (iii).

(4) Polydentate mode with three or more metal centers (iv).

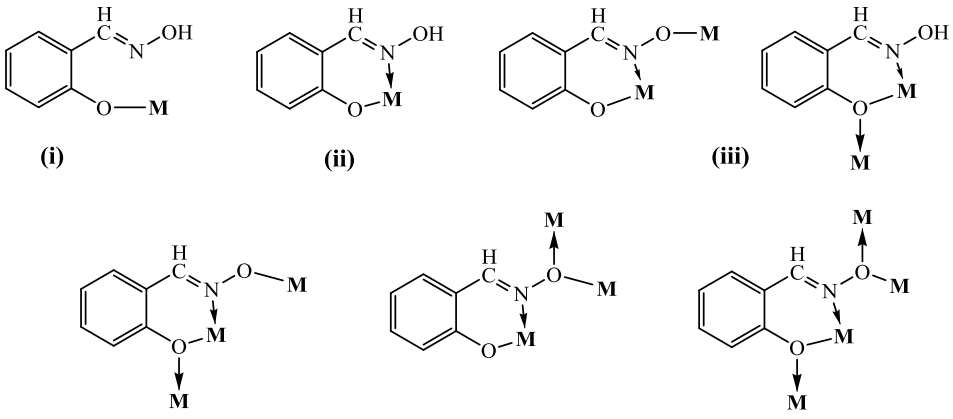


Chart 1: Coordination site of Salicylaldoxime ligand

In this paper, we report the synthesis, structural analyses of a new Pd(II) complexes of salicylaldoxime with amine as co-ligands.

2. Experimental

2.1 General methods and materials

All reactions were carried out in air using standard bench reagents. NMR spectra were recorded at the University of Mashed, Iran on Bruker 500 MHz AVANCE III HD NMR Spectrometer. IR spectra were measured on a Shimadzu FT-IR 8400 spectrophotometer using KBr discs in the range 400–4000 cm^{-1}. Digital molar electric conductivity measurements were recorded on conductivity meter model CD-2005. Elemental analyses were carried out at University of Mashed, Iran. Melting points were measured on a Stuarts SMP10 melting point apparatus and were uncorrected.

2.2 Preparation of $[Pd(Saly)_2]$ (1)

A solution of Salicylaldoxime (HSaly) (0.300 gm, 2.000 mmole) in EtOH (10 mL) with some drops of Et_3N was added with stirring to an aqueous solution of K_2PdCl_4 (0.134gm, 1.00mmole) in (10 mL). A dark yellow ppt. was produced directly. The mixture was stirred for three hours then filtered off and dried in vacuum oven. (Yellow, 0.348 g, 85% yield, m.p (^oC): 269).

2.3 Preparation of $[Pd(Saly)_2(Bipy)]$ (2)

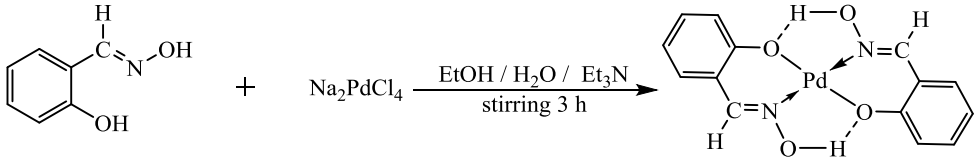
A solution of 2,2'-bipyridyl (Bipy) (0.021gm, 0.132 mmole) in chloroform (5 mL) was added to a yellow solution of $[Pd(Saly)_2]$ (1) (0.050gm, 0.132mmole) in chloroform (10 mL). The mixture was stirred for 3hr. at room temperature. A yellow precipitate produced was filtered off, washed with chloroform and dried in vacuum oven. (Yellow powder, 0.039 g, 54% yield, m.p (^oC): 264-265).

The complexes **$[Pd(Saly)_2(Phen)]$ (3), $[Pd(Saly)_2(en)]$ (4)** were prepared and isolated employing method above.

3. Results and discussion

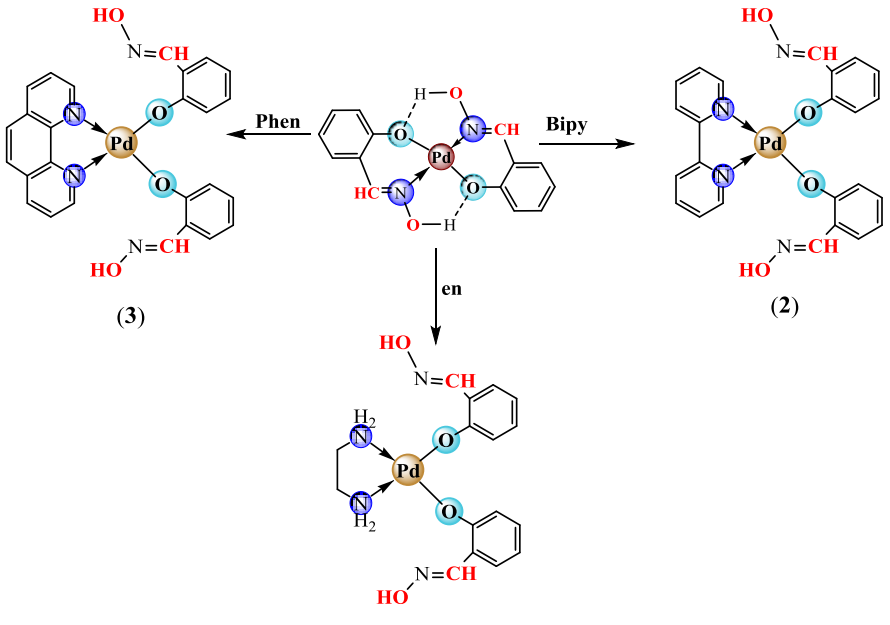
3.1 Synthesis

The reaction of two moles of salicylaldoxime (HSaly) with one equivalent mole of Na_2PdCl_4 in basic medium gave a complex of the type $[Pd(Saly)_2]$(**1**) as a yellow ppt. (**Scheme1**).



(**1**)

Treatment of equivalent molar of amine ligands (amine = Bipy, Phen and en) with $[Pd(Saly)_2]$(**1**) afforded complexes of the formula $[Pd(Saly)_2(amine)]$ (**2-4**). in yield (50-54)% (**Scheme 2**). The complexes are soluble in DMSO and DMF. Additional, its structures were examined by using ^1H, NMR spectra, FT-IR, molar conductivity, and elemental analysis (CHN).



(**3**) (**2**)

(**4**)

Scheme 2: Preparation of complex (2-4)

The molar conductivity measurements of the complexes in DMSO solution (10^{-3}M at 25°C±2) are very low, this indicate that these complexes are non-electrolytic nature[33]. And the CHN analysis and some of the physical properties are listed in the **Table1**.

Table 1: Color, melting point, yield %, molar conductivity and elemental analysis for the Pd(II)-Saly complexes with amine

Seq.	Complexes	Color	Λ in DMSO ($ohm^{-1}. cm^2. mol^{-1}$)	m.p(C^o)	Yield %	Element analysis Found (cal.)%		
						C	H	N
1.	**$[Pd(Saly)_2]$**	**Yellow**	**10.8**	**269**	**85**	**44.40 (44.31)**	**3.19 (3.08)**	**7.40 (7.25)**
2.	**$[Pd(Saly)_2(Bipy)]$**	**Yellow**	**6.8**	**264**	**54**	**53.89 (53,68)**	**3.77 (3.62)**	**10.48 (10.34)**
3.	**$[Pd(Saly)_2(Phen)]$**	**Yellow**	**5.3**	**248**	**50**	**55.88 (55.75)**	**3.61 (3.54)**	**10.02 (9.93)**
4.	**$[Pd(Saly)_2(en)]$**	**Yellow**	**6.2**	**231**	**50**	**43.80 (43.67)**	**4.59 (4.44)**	**12.77 (12.62)**

3.2 Characterization:

3.2.1 ^{1}H NMR spectra

The ^{1}H NMR spectrum of $[Pd(Saly)_2]$ (**Fig. 1**) displayed two singlets at δ8.49ppm and δ10.56ppm, due to the protons of C***H***-N=O and C-N=O***H*** respectively. Also the spectrum displayed two triplet peaks at δ6.74 ppm ($^3J_{H-H}$= 7.88Hz) and δ7.32 ppm ($^3J_{H-H}$= 7.80Hz) assigned to the protons in position ***H***3 and ***H***2, respectively. The ***H***1 and ***H***4 looked as a doublet at δ7.18ppm ($^3J_{H-H}$= 7.90Hz) and δ7.46ppm ($^3J_{H-H}$= 7.80Hz). Each of these signals correspond to 2H.

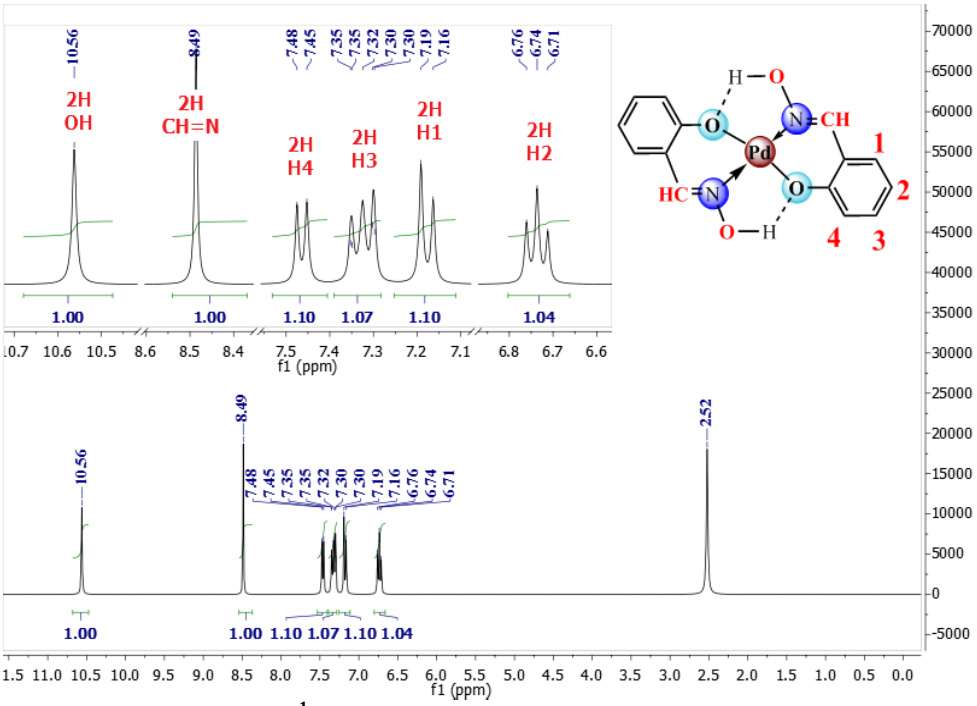


Fig. 1: ^{1}H NMR spectrum of $[Pd(Saly)_2]$ complex

The ^{1}H NMR spectrum of $[Pd(Saly)_2(Bipy)]$ complex (**Fig. 2**) displayed the protons of the Bipy ligand as four separated peaks, three of these peaks as a doublet at δ8.80 ppm, δ8.48 ppm, and δ7.53 ppm due to the protons ***H***5, ***H***7 and ***H***8. While the proton ***H***6 appeared as a triplet peak at δ8.08 ppm. In additional, the spectrum displayed the protons of $Saly^-$ as six separate signals at δ10.54 ppm, and δ8.39 ppm due to C-N=O***H*** and C***H***-N=O, respectively. Also displayed at at δ7.45 ppm (d, 2H), δ7.30 ppm (t, 2H), δ7.16 ppm (d, 2H) and δ6.71 ppm (t, 2H) due to the protons ***H***4, ***H***2, ***H***1 and ***H***3, respectively.

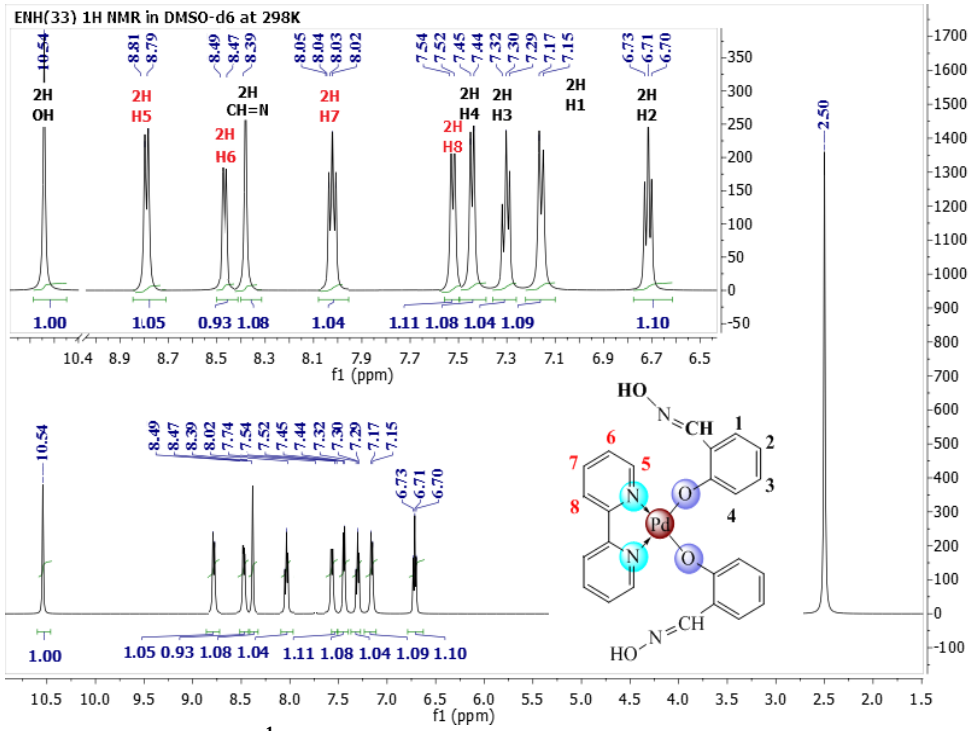


Fig. 2: ^{1}H NMR spectrum of [Pd(Saly)$_2$(Bipy)] complex

The ^{1}H NMR spectrum of [Pd(Saly)$_2$(Phen)] complex (**Fig. 3**) displayed the protons of the Phen ligand as four separated peaks, at δ9.06 ppm (d, 2H, J_{H-H} 7.90Hz), δ8.63 ppm (t, 2H, J_{H-H} 8.00Hz), δ8.06 ppm (s, 2H) and δ7.05 ppm (t, 2H, J_{H-H} 8.00Hz) due to the protons ***H***5, ***H***7, ***H***8 and ***H***6, respectively. Whereas the protons of Saly^{-} ligand appeared as six separated peaks as follows; two singlets at δ10.54ppm and δ8.46ppm, refer to the protons of C-N=O***H*** and C***H***-N=O respectively. Also the spectrum displayed two doublet at δ7.31 ppm ($^{3}J_{H-H}$= 8.00Hz) and δ7.15 ppm ($^{3}J_{H-H}$= 8.00Hz) due to ***H***4 and ***H***1. Whereas ***H***2 and ***H***3 performed as a triplet at δ7.45ppm ($^{3}J_{H-H}$= 8.00Hz) and δ6.72ppm ($^{3}J_{H-H}$= 8.00Hz). Each of these signals correspond to two protons as indicating from the integration values under each peak.

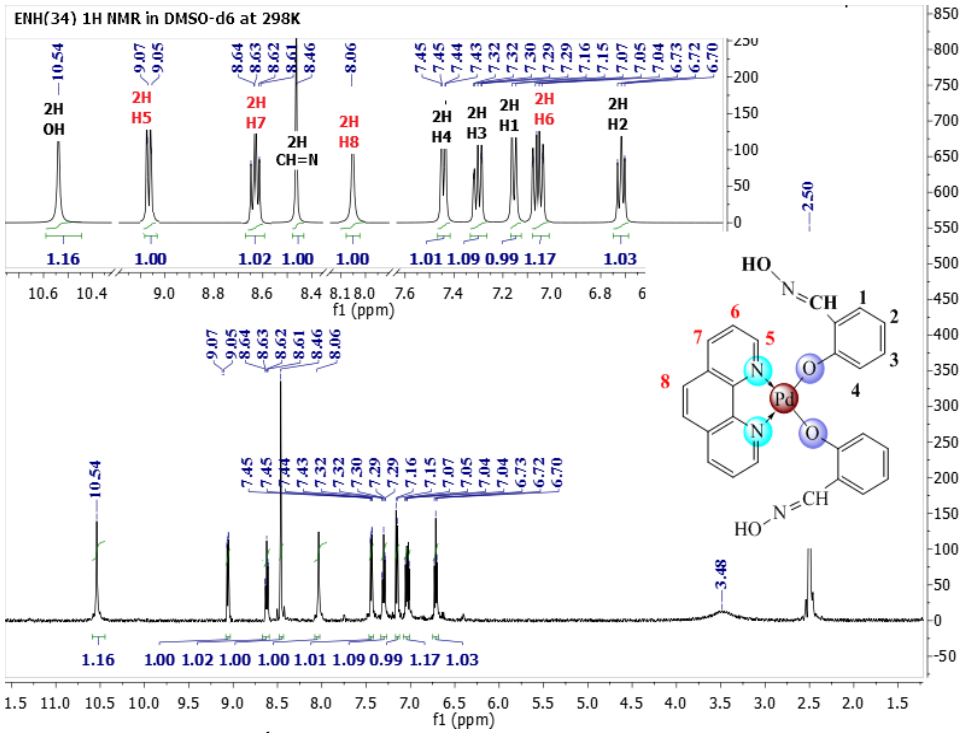


Fig. 2: ^{1}H NMR spectrum of [Pd(Saly)$_2$(Phen)] complex

The ^{1}H NMR spectrum of [Pd(Saly)$_2$(en)] complex (**Fig. 4**) displayed the protons of the ethylene diamine ligand (CH_2 and NH_2) as two singlet peaks, at δ2.78 ppm and δ5.07 ppm, respectively, each of these peaks represent four protons, indicating from the integration values under each peak. In additional the spectrum displayed the protons of the Saly$^-$ liagnd appeared as five signals. The protons of C***H***-N=O and C-N=O***H*** displayed as a singlet at δ8.33ppm andδ 10.22ppm, respectively. And the ***H***4 and ***H***1 displayed as two doublet peaks at δ7.47 ppm ($^{3}J_{H-H}$= 7.5Hz) and δ7.21 ppm ($^{3}J_{H-H}$= 7.8Hz), respectively, Whereas the ***H***2 and ***H***2 displayed as multiplet peak within δ(6.81-6.89ppm).

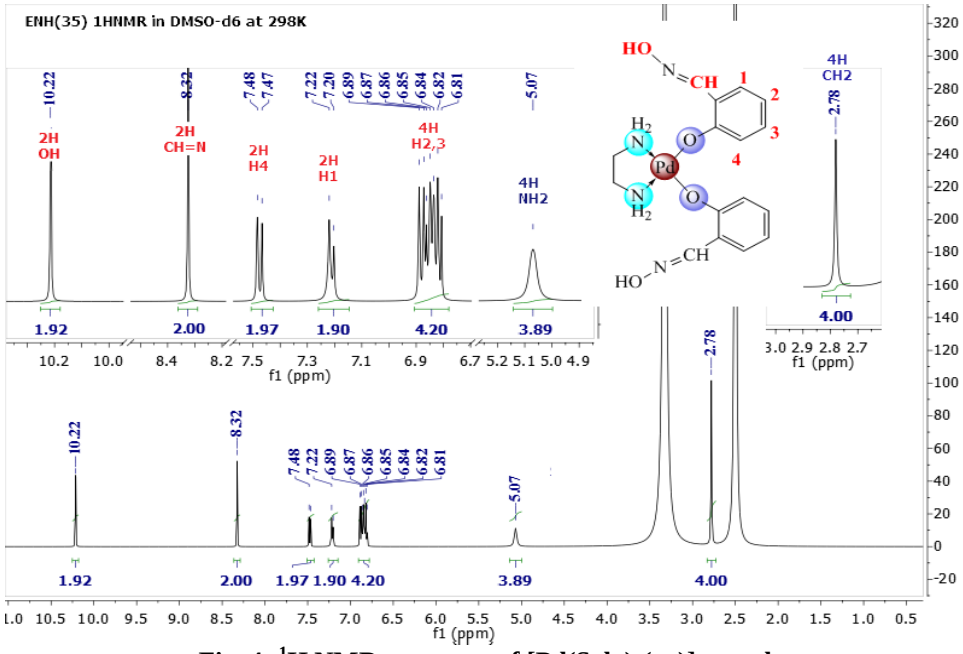


Fig. 4: ^{1}H NMR spectrum of [Pd(Saly)$_2$(en)] complex

Table 2: ^{1}H NMR chemical shifts for the Pd(II)-Saly complexes with amine (ppm).

Seq.	Complexes	δH (ppm)
1	[Pd(Saly)$_2$]	10.56(s, 2H, OH); 8.49 (s, 2H, CH=N); 7.46 (d, 2H, $^{3}J_{HH}$ = 7.80 Hz , H4); 7.32 (t, 2H, $^{3}J_{HH}$ = 7.80 Hz , H3); 7.18 (d, 2H, $^{3}J_{HH}$ = 7.90 Hz , H1); 6.74(t, 2H, $^{3}J_{HH}$ = 7.88 Hz , H2).
2	[Pd(Saly)$_2$(Bipy)]	10.54(s, 2H, OH); 8.80 (d, 2H, $^{3}J_{HH}$ = 8.00 Hz , Bipy-H5); 8.48 (d, 2H, $^{3}J_{HH}$ = 8.00 Hz , Bipy-H6); 8.39 (s, 2H, CH=N); 8.08 (t, 2H, $^{3}J_{HH}$ = 8.00 Hz , Bipy-H7); 7.53 (d, 2H, $^{3}J_{HH}$ = 8.00 Hz , Bipy-H8); 7.44 (d, 2H, $^{3}J_{HH}$ = 7.60 Hz , H4); 7.30 (t, 2H, $^{3}J_{HH}$ = 7.60 Hz , H3); 7.16 (d, 2H, $^{3}J_{HH}$ = 7.60 Hz , H1); 6.71(t, 2H, $^{3}J_{HH}$ = 7.60 Hz , H2).
3	[Pd(Saly)$_2$(Phen)]	10.54(s, 2H, OH); 8.46 (s, 2H, CH=N); 9.06 (d, 2H, $^{3}J_{HH}$ = 8.00 Hz , Phen-H5); 8.63 (d, 2H, $^{3}J_{HH}$ = 8.00 Hz , Phen –H7); 8.06 (s, 2H, $^{3}J_{HH}$ = 8.00 Hz , Phen –H8); 7.45 (t, 2H, $^{3}J_{HH}$ = 8.00 Hz , H4); 7.31 (d, 2H, $^{3}J_{HH}$ = 8.00 Hz , H3); 7.15 (d, 2H, $^{3}J_{HH}$ = 8.00 Hz , H1); 7.05(d, 2H, $^{3}J_{HH}$ = 8.00 Hz , Phen-H6); 6.72(t, 2H, $^{3}J_{HH}$ = 8.00 Hz , H2).
4	[Pd(Saly)$_2$(en)]	10.22(s, 2H, OH); 8.32 (s, 2H, CH=N); 7.47 (d, 2H, $^{3}J_{HH}$ = 7.80 Hz , H4); 7.21 (d, 2H, $^{3}J_{HH}$ = 7.80 Hz , H1); 6.81-6.89 (m, 4H, H2,3); 5.07(bs, 4H, NH_2); 2.78 (s, 4H, CH_2)

3.2.2 IR Spectra

The infra-red spectra of complexes **1-4** are listed in **Table 3** and **Figs. 5-8**. The IR spectrum of free HSaly ligand displayed the υ(Ph-O-H), υ(N-O-H) and υ(CH=N-O) at 3394cm^{-1}, 3373 cm^{-1}, and 1624 cm^{-1}, respectively.

The spectra of prepared complexes showed many characteristic bands, the first new band due to the υ(C=N) of amine ligands (Bipy and Phen) which appeared at (1647-1649) cm^{-1} range, it moved to lower frequency compared with free amines, representing the υ(C=N) contributes in the coordinating with the Pd(II) ion[34-37]. The 2nd and 3rd bands displayed within (3117-3182) cm^{-1} and (1589-1599) cm^{-1} range, due to the υ(N-O-H) and υ(CH=N-O)[4-8]. The 4th band displayed within (1280-1292) cm^{-1} range, refer to the υ(C-O) in the Saly ligand[1-6,26]. The IR spectra displayed a medium to weak intensity band within (466-484) cm^{-1} and (414-416) cm^{-1} range which due to the υ(Pd-O) and υ(Pd-N) modes respectively[38-44]. In addition the spectrum of [Pd(Saly)$_2$(Phen)] complex (**Fig. 7**) displayed distinguishing band at (808)cm^{-1}, due to the C-H out-of-plane deformation vibrations (tetra substituted benzene ring) [35,36]. The IR spectrum of [Pd(Saly)$_2$(en)] complex (**Fig. 8**) displayed two peaks at (3429) cm^{-1} and (3273) cm^{-1} refer to a symmetrical and asymmetrical vibration of NH_2 group [35,36].

Table 3: selected IR bands of the Pd(II)-Saly complexes with amine (cm^{-1}).

Compounds	υ(NO-H)	υ(C-H) Ar.	υ(C-H) Alpha.	υ(C=N) or υ(C-N) (amine)	υ(C=N) (Saly)	υ(N-O)	υ(C-O)	υ(Pd-O)	υ(Pd-N)
Ligand	3373w	3084w	2983w	-	1624s	1259s	993s	-	-
$[Pd(Saly)_2]$	3128w	3049w	2931w	--	1599s	1292s	912s	470w	414
$[Pd(Sal)_2(Bipy)]$	3117w	3047m	2929w	1647 S	1599 s	1288m	910s	466m	414m
$[Pd(Sal)_2(Phen)]$	3126w	3049w	2998w	1649 S	1595 s	1290m	912s	468m	416m
$[Pd(Sal)_2(en)]$	3182m	3055w	2974w	1564s	1593 s	1280m	912s	484m	414m

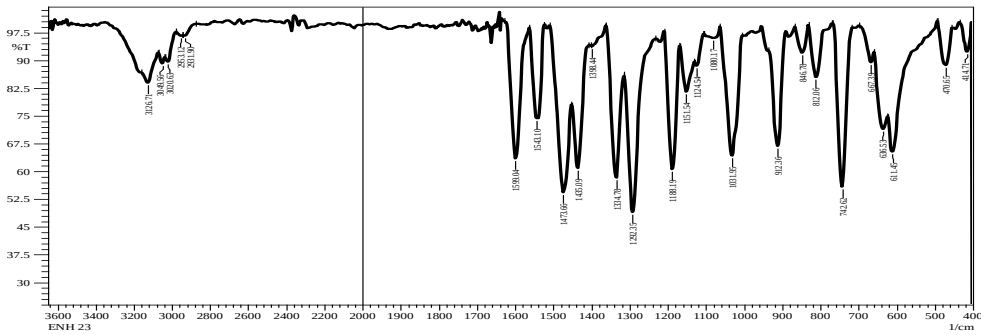


Fig. 5: IR spectrum of $[Pd(Saly)_2]$ complex

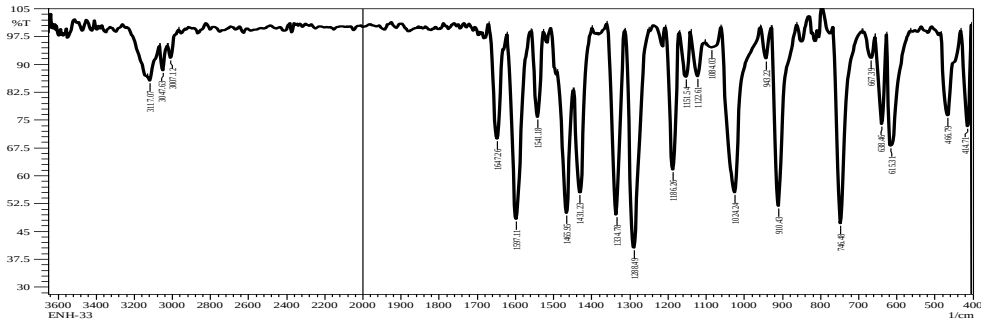


Fig. 6: IR spectrum of $[Pd(Saly)_2(Bipy)]$ complex

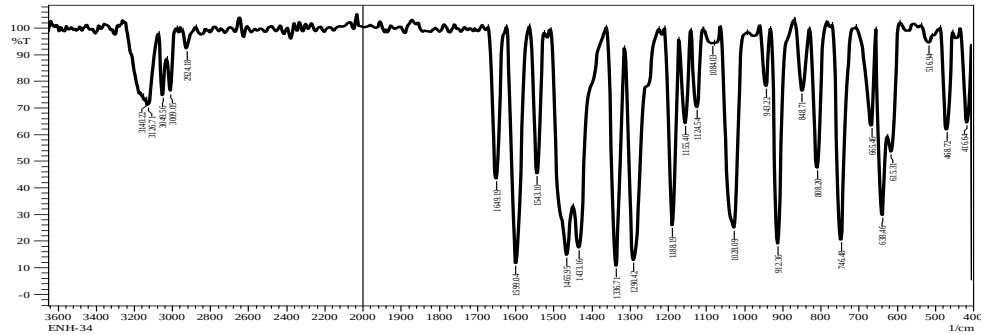


Fig. 7: IR spectrum of $[Pd(Saly)_2(Phen)]$ complex

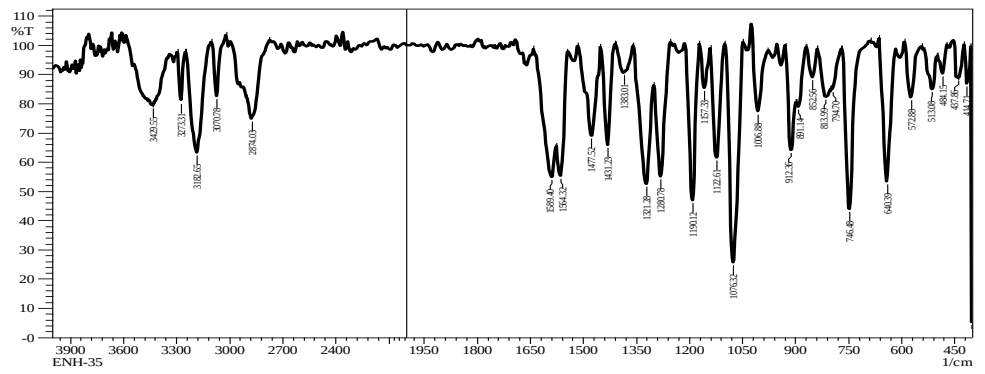


Fig. 8: IR spectrum of $[Pd(Saly)_2(en)]$ complex

Conclusion

The $[Pd(Saly)_2]$ (1), $[Pd(Saly)_2(Bipy)]$ (2), $[Pd(Saly)_2(Phen)]$ (3) and $[Pd(Saly)_2(en)]$ (4) complexes were prepared and characterized. The Salicylaldoximate ligand displayed two coordination modes with the Pd(II) ion, The first mode as bidentate chelating ligand in complex (1), through oxygen atom of hydroxylate group and nitrogen atom of oxime group, while coordinate as monodentate ligand in complexes **(2-4)**, via **O** atom of phenolate group, while the amine ligands coordinated as bi-dentate chelate via the nitrogen in to give a square planner geometry surround the palladium(II) ion.

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تحضير ودراسة طيفية لمعقدات السالسيل الدوكسيم مع ليكاندات الامينيات

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الملخص

حضرت معقدات البلاديوم (II) مع ليكاند السالسيل الدوكسيم مع الامينات كليكاندات مشاركة (البابيريدين، 1،10–فيناثرولين و ثنائي اثيلين امين)، شُخصت المعقدات المحضرة بواسطة التحليل الدقيق للعناصر، الموصلية المولارية الكهربائية، مطيافية الاشعة تحت الحمراء ومطيافية الرنين النووي المغناطيسي للبروتون، حيث أظهرت النتائج ان ليكاند (Saly) يسلك سلوك ليكاند ثنائي السن من خلال ذرة الاوكسجين لمجموعة الهيدروكسيليت الفينولية وذرة نتروجين مجموعة الالدوكسيم، في حين يرتبط بشكل احادي السن من خلال ذرة الاوكسجين لمجموعة الهيدروكسيليت الفينولية في المعقدات (2–4)، لتعطي معقدات ذات بنية مربع مستوي حول أيون البلاديوم (II).