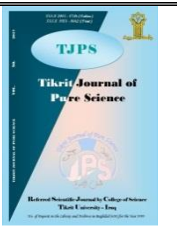
Tikrit Journal of Pure Science
ISSN: 1813 – 1662 (Print) --- E-ISSN: 2415 – 1726 (Online)

Journal Homepage: http://tjps.tu.edu.iq/index.php/j



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

Emad N. Al-Sabawi [1], **Ahmed S. Al-Janabi**[2], **Hayfa Muhammed Jerjes**[1]
[1] *Department of Chemistry, College of Science, Tikrit University, Tikrit, Iraq*
[2] *Department of Biochemistry, College of Veterinary Medicine, Tikrit University, Tikrit, Iraq*

https://doi.org/10.25130/tjps.v26i3.140

ARTICLE INFO.

Article history:

-Received:	**27 / 3 / 2021**
-Received in revised form:	**23 / 4 / 2021**
-Accepted:	**26 / 5 / 2021**
-Final Proofreading:	**1 / 7 / 2021**
-Available online:	**10 / 7 / 2021**

Keywords: Salicylaldoxime; Palladium; oxime; 2,2'-bipyridyl; 1,10-phenanthroline.

Corresponding Author:

Name: Emad N. Al-Sabawi

E-mail:

Tel:

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. However, it was bonded as monodentate ligand in complexes [Pd(Saly)$_2$(amine)] **(2-4)** via the oxygen atom of hydroxylate group. 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. This is attributed to their richly painted complexes with a wide structural variety developed with the majority of transition metals [1-30]. Metal complexes of oxime ligands have arisen 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] and 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).

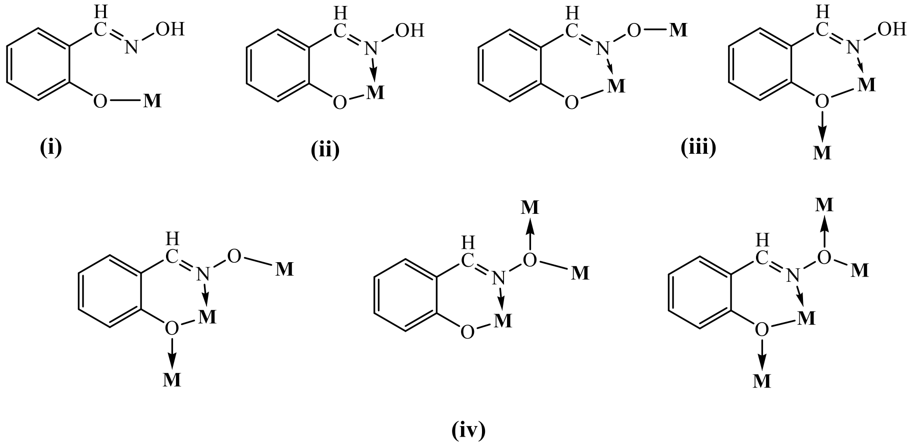


Chart 1: Coordination site of Salicylaldoxime ligand

This paper presents the synthesis, structural analyses of new Pd(II) complexes of salicylaldoxime with amine as co-ligands.

2. Experiment

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 3 hrs. 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)

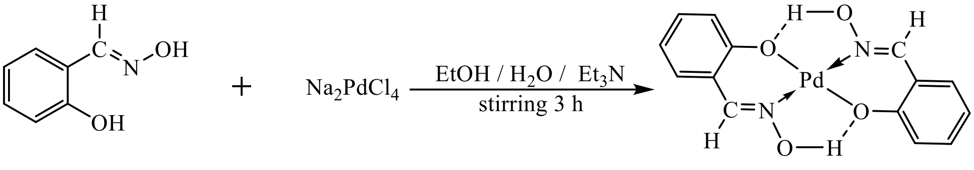
A solution of 2,2'-bipyridyl (Bipy) (0.021gm, 0.132 mmole) in chloroform (5 mL) was added to a yellow solution of $[Pd(Saly)_2]$ (1) (0.050gm, 0.132mmole) in chloroform (10 mL). The mixture was stirred for 3 hrs. at room temperature. A yellow precipitate produced was filtered off, washed with chloroform 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 through employing the method above.

3. Results and Discussion

3.1 Synthesis

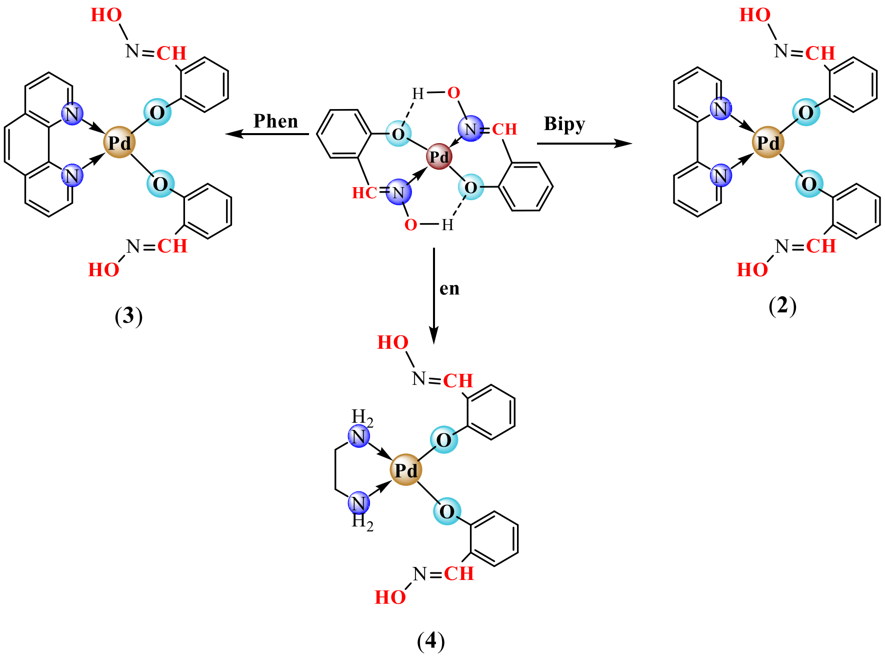
The reaction of two moles of salicylaldoxime (HSaly) with one equivalent mole of Na_2PdCl_4 in basic medium gave a complex of the type $[Pd(Saly)_2]$(**1**) as a yellow ppt. (**Scheme 1**).



(1)

Scheme 1: Preparation of $[Pd(Saly)_2]$(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)% (**Scheme2**). The complexes were soluble in DMSO and DMF. Additionally, their structures were examined by using ^{1}H, NMR spectra, FT-IR, molar conductivity, and elemental analysis (CHN).



Scheme 2: Preparation of complex (2-4)

The molar conductivity measurements of the complexes in DMSO solution (10^{-3}M at 25°C±2) were very low, indicating that these complexes were of a non-electrolytic nature [33]. The CHN analysis and some of the physical properties are listed in **Table 1**.

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)	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 ($^3J_{H\text{-}H}$= 7.88Hz) and δ7.32 ppm ($^3J_{H\text{-}H}$= 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 ($^3J_{H\text{-}H}$= 7.90Hz) and δ7.46ppm ($^3J_{H\text{-}H}$= 7.80Hz). Each of these signals corresponded to 2H.

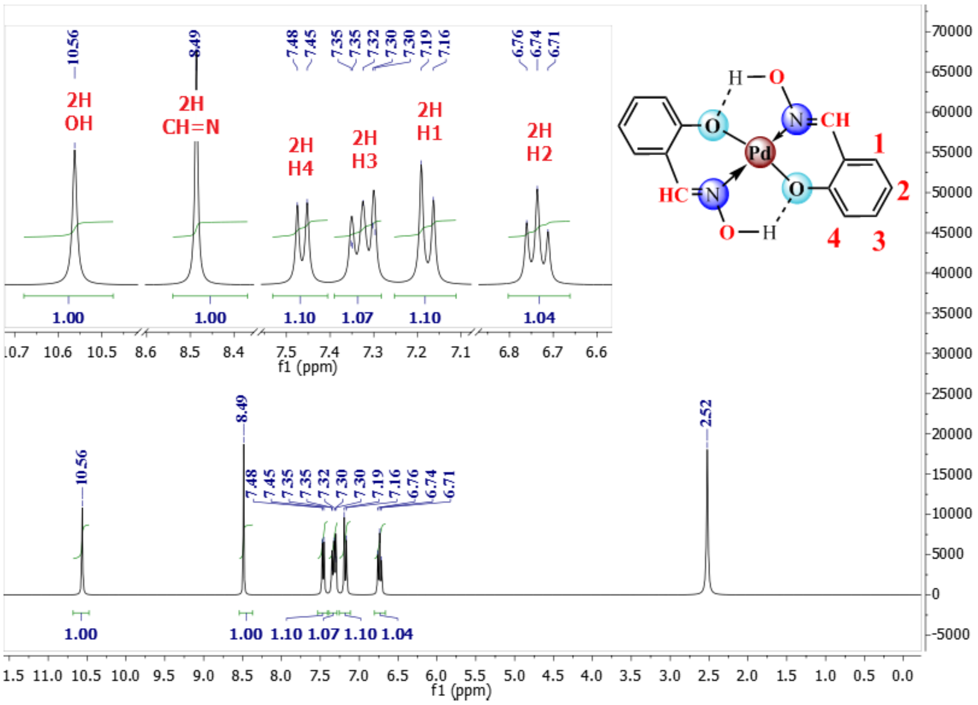


Fig. 1: ^{1}H NMR spectrum of [Pd(Saly)$_2$] complex

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 were shown as a doublet at δ8.80 ppm, δ8.48 ppm, and δ7.53 ppm, due to the protons of H5, H7 and ***H***8. While the proton ***H***6 appeared as a triplet peak at δ8.08 ppm. In addition, the spectrum displayed the protons of Saly$^-$ as six separate signals, two of them were shown at δ10.54 ppm and δ8.39 ppm, due to C-N=O***H*** and C***H***-N=O, respectively. While the other four signals were shown 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 of ***H***4, ***H***2, ***H***1 and ***H***3, respectively.

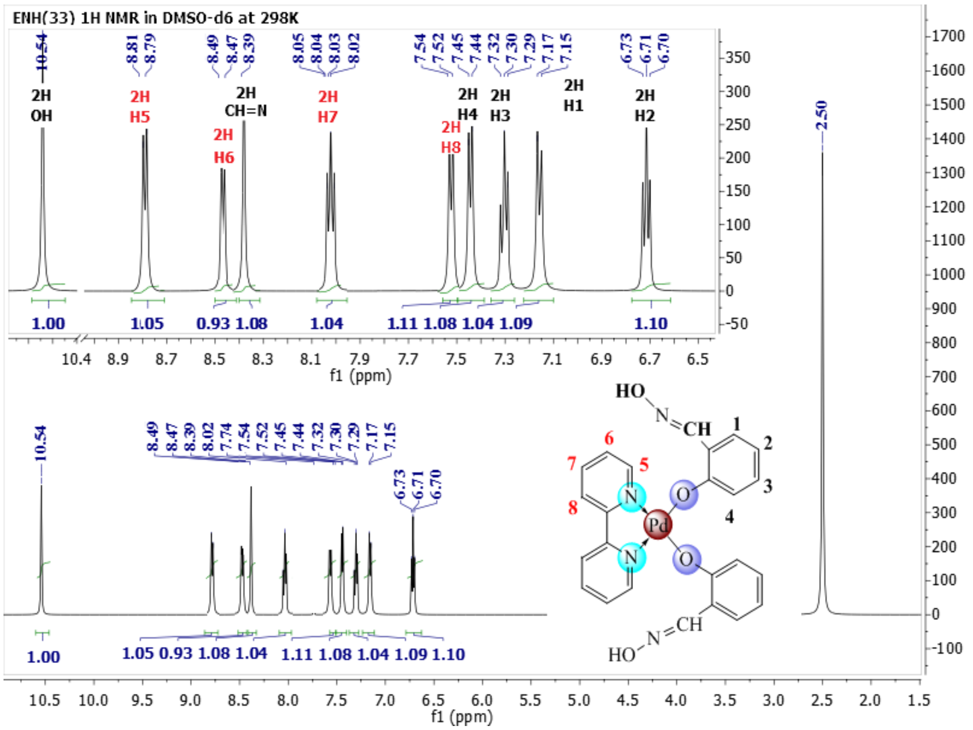


Fig. 2: ^{1}H NMR spectrum of [Pd(Saly)$_2$(Bipy)] complex

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 of ***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, due to the protons of C-N=O***H*** and C***H***-N=O, respectively. Also, the spectrum displayed two doublets at δ7.31 ppm ($^3J_{H-H}$= 8.00Hz) and δ7.15 ppm ($^3J_{H-H}$= 8.00Hz), due to ***H***4 and ***H***1. Whereas ***H***2 and ***H***3 performed as a triplet at δ7.45ppm ($^3J_{H-H}$= 8.00Hz) and δ6.72ppm ($^3J_{H-H}$= 8.00Hz). Each of these signals corresponded to two protons, as indicated from the integration values under each peak.

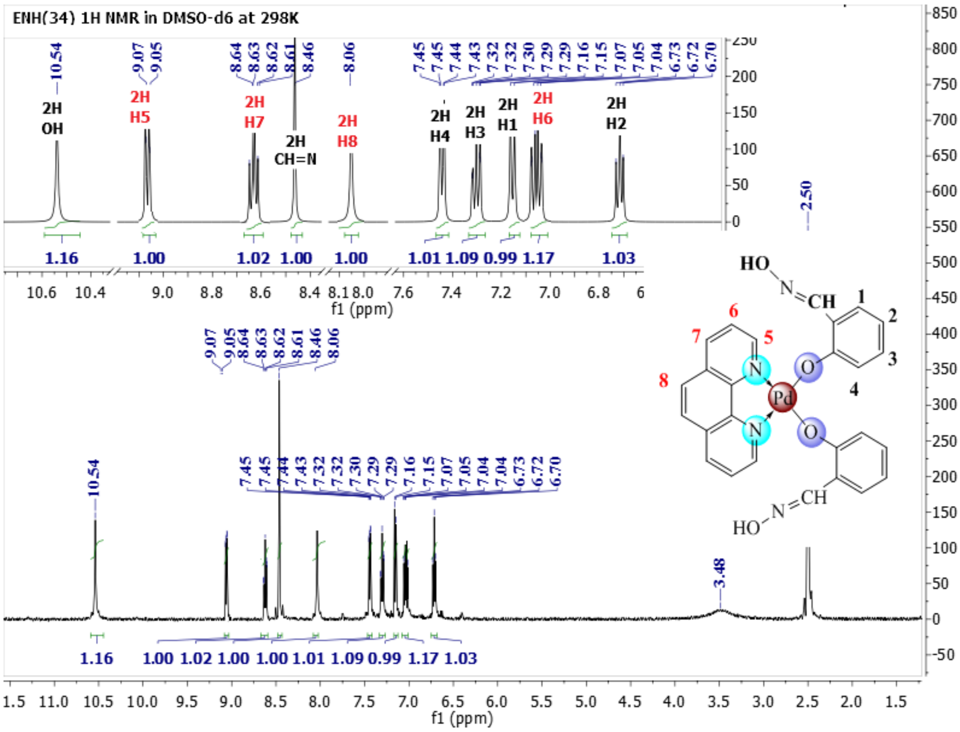


Fig. 3: ^{1}H NMR spectrum of [Pd(Saly)$_2$(Phen)] complex

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 represented four protons, as indicated from the integration values under each peak. In addition, the spectrum displayed the protons of the Saly$^-$ liagnd as five signals. The protons of C**H**-N=O and C-N=O**H** were displayed as a singlet at δ8.33ppm and δ 10.22ppm, respectively. **H**4 and **H**1 were displayed as two doublet peaks at δ7.47 ppm ($^3J_{H\text{-}H}$= 7.5Hz) and δ7.21 ppm ($^3J_{H\text{-}H}$= 7.8Hz), respectively. Whereas **H**2 and **H**3 were displayed as multiplet peak within δ(6.81-6.89ppm).

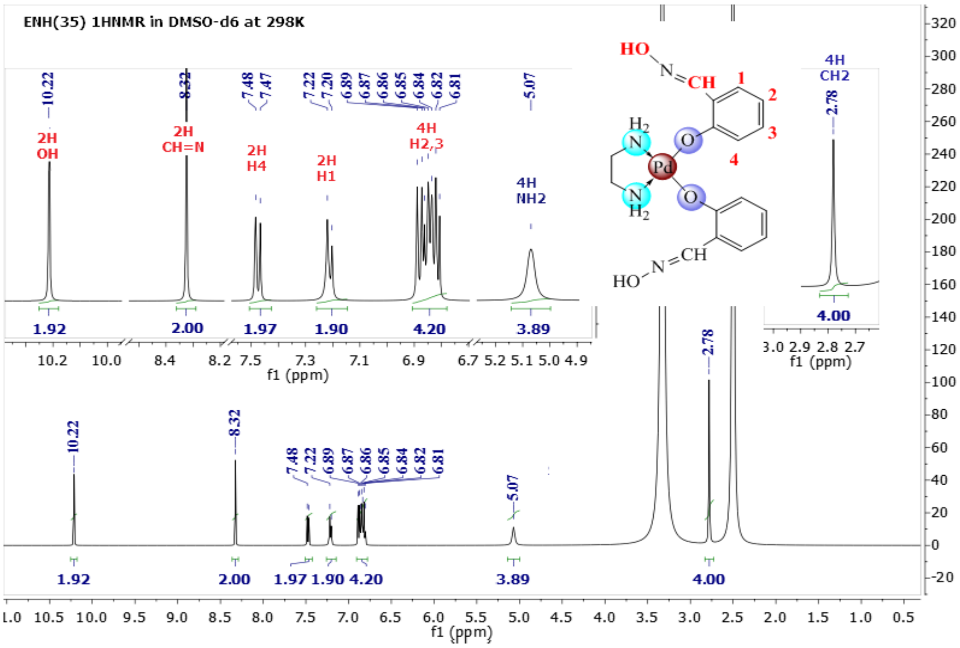


Fig. 4: ^{1}H NMR spectrum of [Pd(Saly)$_2$(en)] complex

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, $^3J_{HH}$ = 7.80 Hz , H4); 7.32 (t, 2H, $^3J_{HH}$ = 7.80 Hz , H3); 7.18 (d, 2H, $^3J_{HH}$ = 7.90 Hz , H1); 6.74(t, 2H, $^3J_{HH}$ = 7.88 Hz , H2).
2	[Pd(Saly)$_2$(Bipy)]	10.54(s, 2H, OH); 8.80 (d, 2H, $^3J_{HH}$ = 8.00 Hz , Bipy-H5); 8.48 (d, 2H, $^3J_{HH}$ = 8.00 Hz , Bipy-H6); 8.39 (s, 2H, CH=N); 8.08 (t, 2H, $^3J_{HH}$ = 8.00 Hz , Bipy-H7); 7.53 (d, 2H, $^3J_{HH}$ = 8.00 Hz , Bipy-H8); 7.44 (d, 2H, $^3J_{HH}$ = 7.60 Hz , H4); 7.30 (t, 2H, $^3J_{HH}$ = 7.60 Hz , H3); 7.16 (d, 2H, $^3J_{HH}$ = 7.60 Hz , H1); 6.71(t, 2H, $^3J_{HH}$ = 7.60 Hz , H2).
3	[Pd(Saly)$_2$(Phen)]	10.54(s, 2H, OH); 8.46 (s, 2H, CH=N); 9.06 (d, 2H, $^3J_{HH}$ = 8.00 Hz , Phen-H5); 8.63 (d, 2H, $^3J_{HH}$ = 8.00 Hz , Phen –H7); 8.06 (s, 2H, $^3J_{HH}$ = 8.00 Hz , Phen –H8); 7.45 (t, 2H, $^3J_{HH}$ = 8.00 Hz , H4); 7.31 (d, 2H, $^3J_{HH}$ = 8.00 Hz , H3); 7.15 (d, 2H, $^3J_{HH}$ = 8.00 Hz , H1); 7.05(d, 2H, $^3J_{HH}$ = 8.00 Hz , Phen-H6); 6.72(t, 2H, $^3J_{HH}$ = 8.00 Hz , H2).
4	[Pd(Saly)$_2$(en)]	10.22(s, 2H, OH); 8.32 (s, 2H, CH=N); 7.47 (d, 2H, $^3J_{HH}$ = 7.80 Hz , H4); 7.21 (d, 2H, $^3J_{HH}$ = 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 was due to υ(C=N) of amine ligands (Bipy and Phen), which appeared at (1647-1649) cm^{-1} range. It moved to a lower frequency compared with free amines, representing the υ(C=N) contribution to the coordination with the Pd(II) ion [34-37]. The 2nd and 3rd bands were displayed within (3117-3182) cm^{-1} and (1589-1599) cm^{-1} range, due to υ(N-O-H) and υ(CH=N-O) [4-8]. The 4th band was displayed within (1280-1292) cm^{-1} range, due to 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} ranges, 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}, due 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)$_{\text{Ar.}}$	υ(C-H)$_{\text{Alpha.}}$	υ(C=N)$_{\text{or}}$ 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

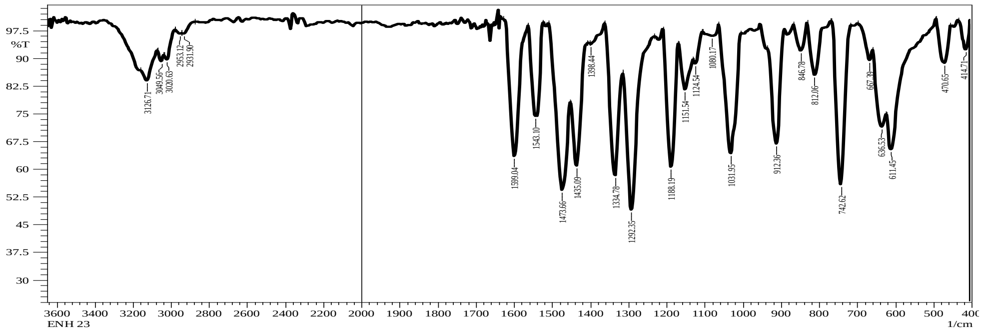


Fig. 5: IR spectrum of [Pd(Saly)$_2$] complex

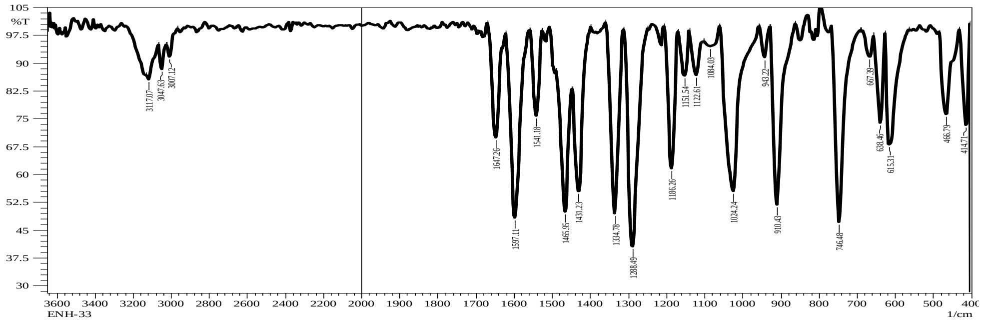


Fig. 6: IR spectrum of [Pd(Saly)$_2$(Bipy)] complex

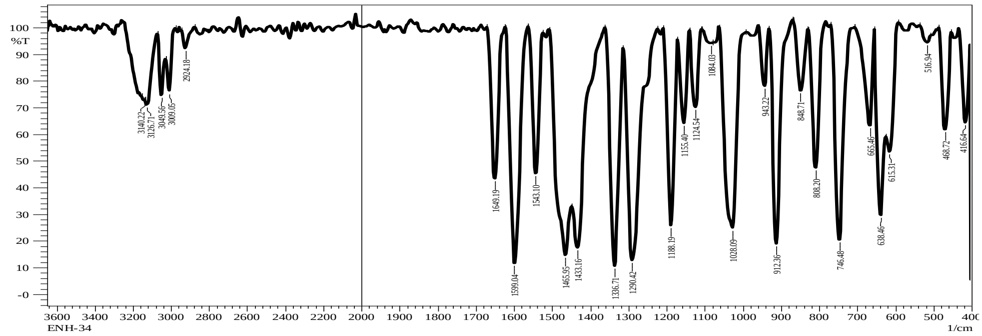


Fig. 7: IR spectrum of [Pd(Saly)$_2$(Phen)] complex

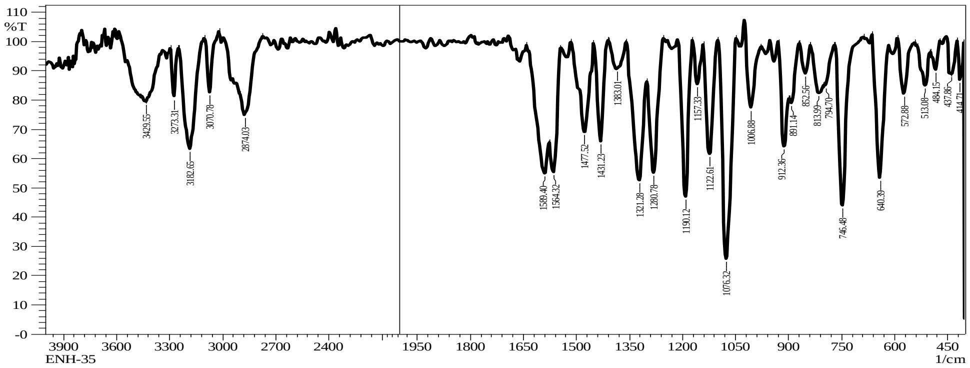


Fig. 8: IR spectrum of [Pd(Saly)$_2$(en)] complex

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 was shown as bidentate chelating ligand in complex (1) through oxygen atom of hydroxylate group and nitrogen atom of oxime group. While it coordinated as monodentate ligand in complexes **(2-4)** via **O** atom of phenolate group. As for the amine ligands, they coordinated as bi-dentate chelate via the nitrogen to give a square planner geometry surrounding the palladium (II) ion.

References

[1] Raptopoulou, C. P., Boudalis, A. K., Lazarou, K. N., Psycharis, V., Panopoulos, N., Fardis, M., ... & Papavassiliou, G.(2008). Salicylaldoxime in manganese (III) carboxylate chemistry: Synthesis, structural characterization and physical studies of hexanuclear and polymeric complexes. *Polyhedron*, *27*(18), 3575-3586.

[2] Ramesh, V., Umasundari, P., & Das, K. K. (1998). Study of bonding characteristics of some new metal complexes of salicylaldoxime (SALO) and its derivatives by far infrared and UV spectroscopy. *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy*, *54*(2), 285-297.

[3] Jayaraju, D., & Kondapi, A. K. (2001). Anti-cancer copper salicylaldoxime complex inhibits topoisomerase II catalytic activity. *Current Science*, 787-792.

[4] Ramesh, V., Umasundari, P., & Das, K. K. (1998). Study of bonding characteristics of some new metal complexes of salicylaldoxime (SALO) and its derivatives by far infrared and UV spectroscopy. *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy*, *54*(2), 285-297.

[5] Burger, K., & Egyed, I. (1965). Some theoretical and practical problems in the use of organic reagents in chemical analysis—V: Effect of electrophilic and nucleophilic substituents on the stability of salicylaldoxime complexes of transition metals. *Journal of Inorganic and Nuclear Chemistry*, *27*(11), 2361-2370.

[6] Wenzel, M., Forgan, R. S., Faure, A., Mason, K., Tasker, P. A., Piligkos, S. & Plieger, P. G. (2009). A New polynuclear coordination type for (salicylaldoxime) copper (II) complexes: Structure and magnetic properties of an (oxime) Cu6 Cluster.

[7] Aggarwal, R. C., Singh, N. K., & Singh, R. P. (1984). Magnetic and spectroscopic studies on Salicylaldoxime and o-Hydhoxynaphthaldoxime complexes of some divalent 3d metal ions. *Synthesis and Reactivity in Inorganic and Metal-Organic Chemistry*, *14*(5), 637-650.

[8] Smith, A. G., Tasker, P. A., & White, D. J. (2003). The structures of phenolic oximes and their complexes. *Coordination chemistry reviews*, *241*(1-2), 61-85.

[9] Cupertino, D., McPartlin, M., & Zissimos, A. M. (2001). Synthesis of cobalt (II) complexes of derivatised salicylaldoxime ligands; X-ray crystal structures of DMSO adducts of bis (3-nitro-5-methylsalicylaldoximato) cobalt (II) and bis (3-nitro-5-phenylsalicylaldoximato) cobalt (II). *Polyhedron*, *20*(26-27), 3239-3247.

[10] Abualhaija, M. M., & van den Berg, C. M. (2014). Chemical speciation of iron in seawater using catalytic cathodic stripping voltammetry with ligand competition against salicylaldoxime. *Marine Chemistry*, *164*, 60-74.

[11] Ma, Y., Zhang, W., Ou-Yang, Y., Yoshimura, K., Liao, D. Z., Jiang, Z. H., & Yan, S. P. (2007). A three-spin complex [Ni (salox) 2 (IM4Py) 2] containing salicylaldoxime and nitronyl nitroxide: Structure and magnetic properties. *Journal of molecular structure*, *833*(1-3), 98-101.

[12] Kumar, B., Prasad, K. K., & Srivastawa, S. K. (2010). Synthesis of oxygen bridged complexes of Cu (II) or Ni (II)-salicylaldoxime with alkali metal salts of some organic acids and studies on their antimicrobial activities. *Oriental Journal of Chemistry*, *26*(4), 1413.

[13] Lumme, P., Elo, H., & Jänne, J. (1984). Antitumor activity and metal complexes of the first transition series: Trans-bis (salicylaldoximato) copper (II) and related copper (II) complexes, a novel group of potential antitumor agents. *Inorganica chimica acta*, *92*(4), 241-251.

[14] Thorpe, J. M., Beddoes, R. L., Collison, D., Garner, C. D., Helliwell, M., Holmes, J. M., & Tasker, P. A. (1999). Surface coordination chemistry: Corrosion inhibition by tetranuclear cluster formation of iron with salicylaldoxime. *Angewandte Chemie International Edition*, *38*(8), 1119-1121.

[15] Prasad, R. L., Aggarwal, R. C., & Bala, R. (1990). Ternary complexes of 3d metal (II) ions with acetylacetone and salicylaldoxime. *Transition Metal Chemistry*, *15*(5), 379-382.

[16] Birnara, C., Kessler, V. G., & Papaefstathiou, G. S. (2009). Mononuclear gallium (III) complexes based on salicylaldoximes: Synthesis, structure and spectroscopic characterization. *Polyhedron*, *28*(15), 3291-3297.

[17] Belkhettab, I., Boutamine, S., Slaouti, H., Zid, M. F., Boughzala, H., & Hank, Z. (2020). Synthesis, characterization and structural study of new vanadium complexes with phenolic oxime ligands. *Journal of Molecular Structure*, *1206*, 127597.

[18] Nakamura, H., Shimura, Y., & Tsuchida, R. (1963). A study of metal complexes of analytical importance: II complexes of Salicylaldoxime and C-Methyl-salicylaldoxime with Vanadium (V). *Bulletin of the Chemical Society of Japan*, *36*(3), 296-301.

[19] Jayaraju, D., Gopal, Y. V., & Kondapi, A. K. (1999). Topoisomerase II is a cellular target for antiproliferative cobalt salicylaldoxime complex. *Archives of biochemistry and biophysics*, *369*(1), 68-77.

[20] Das, A. K. (1990). Statistical aspects of the stabilities of ternary complexes of cobalt (II), nickel (II), copper (II) and zinc (II) involving aminopolycarboxylic acids as primary ligands and salicylaldoxime as a secondary ligand. *Transition Metal Chemistry*, *15*(1), 75-77.

[21] Pannu, A. P. S., Stevens, J. R., & Plieger, P. G. (2013). Aryl-Linked Salicylaldoxime-Based Copper (II) Helicates and "Boxes": Synthesis, X-ray analysis, and anion influence on complex structure. *Inorganic chemistry*, *52*(16), 9327-9337.

[20] Bokach, N. A., Haukka, M., Pombeiro, A. J., Morozkina, S. N., & Kukushkin, V. Y. (2002). Concentration dependent switch from addition to substitution in the reaction between salicylaldoxime and a nitrile platinum (IV) complex. *Inorganica chimica acta*, *336*, 95-100.

[23] Li, Z., Rao, F., Song, S., Uribe-Salas, A., & López-Valdivieso, A. (2019). Effects of common ions on adsorption and flotation of malachite with salicylaldoxime. *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, *577*, 421-428.

[24] Gass, I. A., Milios, C. J., Collins, A., White, F. J., Budd, L., Parsons, S.,. & Brechin, E. K. (2008). Polymetallic clusters of iron (III) with derivatised salicylaldoximes. *Dalton Transactions*, (15), 2043-2053.

[25] Prasad, R. L., Bala, R., & Aggarwal, R. C. (1987). Synthesis and spectroscopic studies on some 3d metal (II) hetero ligand complexes of Salicylaldoxime and 1-Nitroso-2-Naphthol. *Synthesis and Reactivity in Inorganic and Metal-Organic Chemistry*, *17*(7), 709-722.

[26] Kukushkin, V. Y., & Pombeiro, A. J. (1999). Oxime and oximate metal complexes: Unconventional synthesis and reactivity. *Coordination Chemistry Reviews*, *181*(1), 147-175.

[27] Babu, M. S., Reddy, K. H., & Krishna, P. G. (2007). Synthesis, characterization, DNA interaction and cleavage activity of new mixed ligand copper (II) complexes with heterocyclic bases. *Polyhedron*, *26*(3), 572-580.

[28] Ma, Y., Zhang, W., Xu, G. F., Yoshimura, K., Liao, D. Z., Jiang, Z. H., & Yan, S. P. (2007). Synthesis, structure and magnetic properties of a novel nickel (II) radical heterospin complex with salicylaldoxime. *Zeitschrift für anorganische und allgemeine Chemie*, *633*(4), 657-660.

[29] Biefeld, L. P., & Howe, D. E. (1939). Separation and determination of copper and nickel bysalicylaldoxime. *Industrial & Engineering Chemistry Analytical Edition*, *11*(5), 251-253.

[30] Mason, K., Gass, I. A., White, F. J., Papaefstathiou, G. S., Brechin, E. K., & Tasker, P. A. (2011). Hexa-and octanuclear iron (III) salicylaldoxime clusters. *Dalton Transactions*, *40*(12), 2875-2881.

[31] Kukushkin, Y. N., Krylov, V. K., Kaplan, S. F., Calligaris, M., Zangrando, E., Pombeiro, A. J., & Kukushkin, V. Y. (1999). Different chlorination modes of oximes: Chlorination of salicylaldoxime coordinated to platinum. *Inorganica chimica acta*, *285*(1), 116-121.

[32] Vogel, A. I. (1963). *A textbook of quantitative inorganic analysis*. London: Longmans.

[33] Geary, W.J. (1971). The use of conductivity measurements in organic solvents for the characterization of coordination compounds. *Coordination Chemistry Reviews*, **7** **(1)**: 81–122

[34] Al-Janabi, A. S., Al-Jumaili, W. A., Al-Hayaly, L. J., Al-Jibori, S. A., Schmidt, H., Wagner, C., & Hogarth, G. (2020). Synthesis and in vitro cytotoxicity studies of Pd (II) and Pt (II) acetamide complexes: Molecular structures of trans-[PdCl2 (bzmta)2]. DMF (bzmta= 2-acetylamino-6-methylbenzothiazole) and cis-[PtCl2 (bzta) 2]. 2DMF (bzta= 2-acetylaminobenzothiazole). *Polyhedron*, *185*, 114591

[35] Al-Janabi, A. S., Al-Dulaimi, A. A., Gergees, H. M., & Saleh, M. H. (2019). Synthesis and spectroscopic studies of new palladium (II) complexes of N-hydroxymethysacharin (Sac-CH2OH) and amine or diamines ligands. *Oriental Journal of Chemistry*, *35*(1), 186.

[36] Al-Janabi, A. S. (2016). Synthesis and characterization of Pd (II) and Pt (II) complexes containing mixed ligands of thione and diamine. *Tikrit Journal of Pure Science*, *21*(7).

[37] Al-Jibori, S. A., Al-Janabi, A. S., Al-Sahan, S. W., & Wagner, C. (2021). Pd (II)-pyrrolidine dithiocarbamate complexes: Synthesis, spectroscopic studies and molecular structure of [Pd (PyDT)(ppy)]. *Journal of Molecular Structure*, *1227*, 129524.

[38] Al-Janabi, A. S., Al-Samrai, O. A. A., & Yousef, T. A. (2020). New palladium (II) complexes with 1-phenyl-1H-tetrazole-5-thiol and diphosphine: Synthesis, characterization, biological, theoretical calculations and molecular docking studies. *Applied Organometallic Chemistry*, *34*(12), e5967.

[39] Al-Janabi, A. S., Irzoqi, A. A., & Ahmed, S. A. (2018). Synthesis and characterization of mixed ligands cadmium (II) complexes with N-Hydroxymethylsaccharinate and diphosphines. *Tikrit Journal of Pure Science*, *21*(3), 54-60.

[40] Al-Jibori, A. A., Al-Jibori, S. A., & Al-Janabi, A. S. (2019). Palladium (II) and platinum (II) mixed ligand complexes of metronidazole and saccharinate or benzisothiazolinonate ligands, synthesis and spectroscopic investigation. *Tikrit Journal of Pure Science*, *24*(6), 26-32.

[41] Al-Jibori, S. A., Al-Nassiry, A. I., Al-Janabi, A. S., & Al-Hayaly, L. J. (2020). Synthesis and

characterization of platinum (II) and palladium (II) diphosphine complexes with heterocyclic N-acetamide or saccharinate ligands. *Chemical Data Collections*, *30*, 100542.

[42] Al-Jibori, M. H., Buttrus, N. H., & Al-Janabi, A. S. (2018). Synthesis and studies Pd (II)-NHC complexes with thiosaccharinate, saccharinate or benzothiazolinate ligands. *Tikrit Journal of Pure Science*, *22*(2), 99-103.

[43] Al-Janabi, A. S. (2017). In vitro antimicrobial studies of new Zn (II) complexes of N-hydroxymethylsaccharin (Sac-CH2OH) and amine ligands. *Journal of Kerbala for Agricultural Sciences*, *4*(5), 140-149.

[44] Al-Janabi, A. S., Zaky, R., Yousef, T. A., Nomi, B. S., & Shaaban, S. (2020). Synthesis, characterization, computational simulation, biological and anticancer evaluation of Pd (II), Pt (II), Zn (II), Cd (II), and Hg (II) complexes with 2-amino-4-phenyl-5-selenocyanatothiazol ligand. *Journal of the Chinese Chemical Society*, *67*(6), 1032-1044.

تحضير ودراسة طيفية لمعقدات السالسيل الدوكسيم مع ليكاندات الامينيات

عماد ناصر السبعاوي[1] ، احمد شاكر الجنابي[2]، هيفاء محمد جرجيس[1]

[1] قسم الكيمياء ، كلية العلوم ، جامعة تكريت ، تكريت، العراق

[2] فرع الكيمياء الحياتية ، كلية الطب البيطري، جامعة تكريت، تكريت ، العراق

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).