



New Salicylanilide-Hg(II) complexes with phosphine ligands, Synthesis and spectroscopic investigation

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ABSTRACT

A new complex of the type $[\text{Hg}(\kappa^2\text{-Saln})_2]$ (**1**) was prepared from the reaction of mercury acetate and salicylanilide (HSaln) in (1:2) molar ratio EtOH as a solvent with presence an Et_3N as a base. Treatment of complex (**1**) with diphosphine {where diphos: dppe, dppp, dppb dppf and dppmS_2 } in (1:1) (complex: diphos) molar ratio afforded a complexes of the types $[\text{Hg}(\kappa^1\text{-Saln})_2(\text{diphos})]$ (**2-6**), where as a complexes of the types $[\text{Hg}(\kappa^1\text{-Saln})_2(\text{phos})_2]$ (**7,8**) when treatment of complex(**1**) with two moles of monophosphine (PPh_3 or SPPH_3). The prepared complexes were fully characterization by many technical such as elemental analysis, molar conductivity, infrared spectroscopy, and nuclear magnetic resonance (^1H and ^{31}P). In complex (**1**) the (Saln^-) was bonded as bidentate chelating ligand through the oxygen atoms of the carbonyl and deprotonated hydroxyl group, whereas bonded as monodentate ligand through the oxygen atom of deprotonated hydroxyl group in all other complexes (**2-8**). The geometry of the complexes (**1-8**) is a tetrahedral around the mercury (II) ion.

1. Introduction

Salicylanilide {2-Hydroxy-N-phenylbenzamide; or 2-Hydroxybenzanilide, or N-phenylsalicylamide, (HSaln)} is known as a fungicides for topical uses and antimicrobial agents in soaps[1,2]. The salicylanilide compounds have been widely used to treat intestinal tapeworm, hydatid diseases, schistosomiasis, liver fluke infection in human and animals[3]. In veterinary medicine, the salicylanilides derivatives (such as niclosamide, tereanol, oxclozanide, rafoxanide, and clioxanide) are used to treat tapeworm and fluke infections from domestic animals [4-8]. HSaln ligand can be showed different coordination fashion to metal ions, being act as monodentate fashion through the oxygen atom of deprotonated hydroxyl group, or through the neutral oxygen of hydroxyl or carbonyl group, and it also has the ability to bonding as a bidentate fashion through oxygen and nitrogen donor atom [9]. Complexes of salicylanilide or its derivatives with metal ions such as Pd(II), Pt(II), Mo(V), Os(VI), and other ions have been recently been synthesized and characterized [10-14]. Herein, we describe the preparation of mixed ligand Hg(II) salicylanilidate (Saln^-) complexes with

phosphine. These prepared complexes were investigated by spectroscopic and physical methods.

2. Experimental

2.1 Materials and Methods:

All chemical compounds ($\text{Hg}(\text{oAc})_2 \cdot x\text{H}_2\text{O}$, salicylanilide, phosphines, EtOH, and CHCl_3) were supplied and used without further purification. IR spectra were recorded on Shimadzu 8400 S FTIR spectrophotometer as KBr disc in (4000-400) cm^{-1} range. All NMR spectra (^1H and ^{31}P - $\{^1\text{H}\}$) were recorded on Bruker av 400 NMR spectrometer in DMSO-d_6 . The conductivity measurement of the prepared complexes solution (10^{-3}M) in DMSO were measured on Digital conductivity meter CD 2005. Melting points were recorded on SMP40 - Stuart company and were uncorrected.

2.2. Preparation of $[\text{Hg}(\text{Saln})_2]$ (**1**)

An ethanolic solution of HSaln (1.338g; 6.274mmole) in (15ml) was added to a suspension of $\text{Hg}(\text{oAc})_2 \cdot x\text{H}_2\text{O}$ (1.000g; 3.137mmole) in EtOH (10ml), with some drops of Et_3N as a base, a clear light brown solution was formed. The mixture was refluxed for 1.5 hr, and filtered off and left aside for evaporation at room temperature. The gum product

was washed several times with diethyl ether, and the light brown ppt formed was collected and dried under vacuum (Yield : 0.1915g ; 98% ; m.p °C: (80-82)).

2.2.2 Preparation of [Hg(Saln)₂(dppe)] (2)

A solution of Bis(diphenylphosphino)ethane (dppe) (0.050g; 0.128mmole) in CHCl₃ (10ml) was added to a solution of [Hg(Saln)₂] (0.080g; 0.128mmole) in CHCl₃ (10ml) with stirring. The mixture was stirring for 2hr at room temperature, The creamy solution was filtered and left aside to evaporate the solvent. The gum product was washed off with diethyl ether and the white ppt. was collected and dried in oven under vacuum .(Yield : 0.1001g; 76% ; m.p °C: (92-93)).

2.2.3 preparation of [Hg(Saln)₂(dppp)] (3)

To solution of complex (1) (0.080g; 0.128mmole) in CHCl₃ (10ml), a solution of Bis (diphenylphosphino) propane (dppp) (0.055g; 0.128mmole) in CHCl₃ was added with stirring. The color of solution was changed to off white. The solution was stirred at room temperature for 2hr and filtered off and left aside for slow evaporation at room temperature. The gum product was washed with diethyl ether for several times to formed off white ppt. and the was collected and dried in oven under vacuum . (Yield :0.067g; 50% ; m.p °C: (72-74)).

The [Hg(Saln)₂(dppb)] (4) ; [Hg(Saln)₂(dppf)] (5) and [Hg(Saln)₂(dppmS₂)] (6) complexes was prepared and isolated in a similar method.

2.2.4 preparation of [Hg(Saln)₂(SPPH₃)₂] (7)

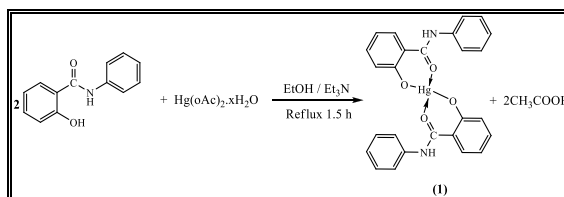
A solution of triphenyl phosphine sulfide (0.075g; 0.256mmole) in CHCl₃ (10ml) was added to a solution of [Hg(Saln)₂] (0.080g; 0.128mmole) in CHCl₃ (10ml) with stirring. The color of solution was changed to a light brown. The mixture was stirring for 3hr at room temperature, and filtered off, and left aside for slow evaporation. The gum product was formed and washed with diethyl ether for several times and the light brown ppt. was collected and dried

.(Yield : 0.041g ; 26% ; m.p °C: (103-104)). The [Hg(Saln)₂(PPh₃)₂] (8) complex was prepared and isolated in a similar method.

3. Results and Discussion

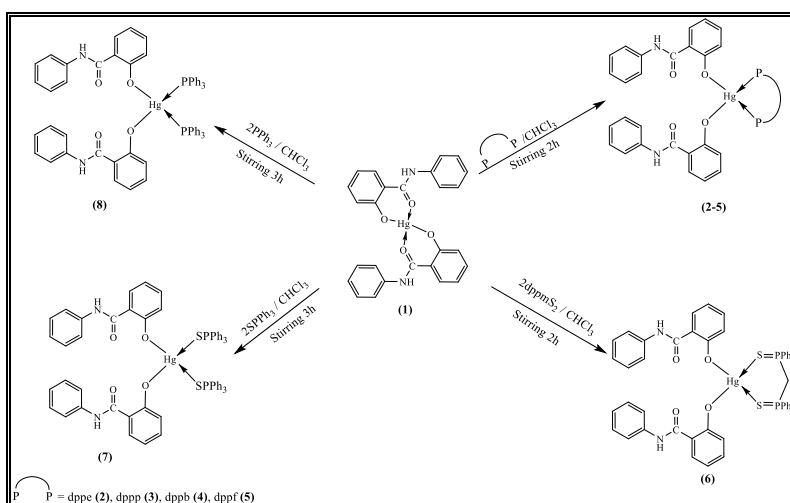
3.1 Synthesis

The reaction of Salicylanilide (HSaln) with mercury(II) acetate in (2:1) (ligand : metal) molar ratio, in absolute ethanol with some drops of Et₃N as a base , gave a light brown complex of the type [Hg(κ²-Saln)₂] (1) as a solo product (scheme 1). The Saln⁻ ligand was coordinated as bidentate chelating ligand through the oxygen atoms of carbonyl group and deprotonated hydroxyl group to afford a tetrahedral geometry around the central atom ion.



Scheme 1: preparation of complex (1)

The treatment of complex (1) with diphosphine (diphos= dppe , dppp , dppb , dppf and dppmS₂) in equal molar ratio in chloroform as a solvent afford a complexes of the type [Hg(κ¹-Saln)₂(κ²-diphos)] (2-6) (See Scheme 2). The (Saln⁻) was bonded through oxygen atom of the deprotonated hydroxyl group as a monodentate ligand, whereas the diphosphine ligands were bonded as bidentate chelating ligand. One mole of the complex (1) was reacted with two moles of triphenyl phosphine or triphenyl phosphine sulfide to afford a complexes of the types [Hg(κ¹-Saln)₂(phos)₂] (7-8) (see scheme 2). The Saln⁻ ligand behave as monodentate ligand through the oxygen atom of the deprotonated hydroxyl group.



Scheme 2: preparation of complexes (2-9).

All prepared complexes are air stable in solid state, and insoluble in diethyl ether, hexane, methanol and distilled water but soluble in chloroform, dichloromethane, DMSO and DMF. The elemental

analysis are listed in Table 1 and they agreed with theoretical analysis. The conductivity of freshly prepared complexes solutions were measured in DMSO at room temperature and that the complexes

are non-electrolyte [15]. The prepared complexes were characterization by many technical CHN analysis, molar conductivity, IR, NMR spectroscopy

(^1H and, ^{31}P). The CHN analysis, physical properties, IR and NMR data are listed in Table (1 to 3).

Table 1. Color, yield, m.p.(°C), and elemental analysis of prepared complexes (1-8)

Seq.	Complexes	Color	m.p.(°C)	Yield %	Λ (Ohm $^{-1}$.cm 2 .mol $^{-1}$)	Elemental analysis Found(cal.) %		
						C	H	N
1	[Hg(Saln) $_2$]	light brown	80-82	98	4.12	50.23 (49.96)	3.62 (3.23)	4.95 (4.48)
2	[Hg(Saln) $_2$ (dppe)]	White	92-93	76	10.90	61.38 (61.02)	4.59 (4.33)	3.07 (2.74)
3	[Hg(Saln) $_2$ (dppp)]	White	72-74	50	10.5	61.52 (6.36)	4.81 (4.47)	2.94 (2.70)
4	[Hg(Saln) $_2$ (dppb)]	White	95-97	61	11.2	62.01 (61.68)	4.89 (4.60)	3.10 (2.66)
5	[Hg(Saln) $_2$ (dppf)]	White	119-120	47	10.34	----	----	---
6	[Hg(Saln) $_2$ (dppmS $_2$)]	Brown	128-130	49	3.90	----	----	----
7	[Hg(Saln) $_2$ (SPPH $_3$) $_2$]	Light brown	103-104	26	2.90	61.64 (61.35)	4.38 (4.15)	2.51 (2.13)
8	[Hg(Saln) $_2$ (PPh $_3$) $_2$]	Light brown	59-61	93	11.73	65.01 (64.78)	4.89 (4.38)	2.87 (2.44)

3.2 Spectroscopic studies

3.2.1 Infrared spectra

The IR selected bands of salicylanilide and its complexes are listed in **Table 2** and **Fig 1** and **2**. The IR spectrum of free salicylanilide showed bands at (3302), (3148), (1618) and (1558) cm $^{-1}$ which due to $\nu(\text{NH})$; $\nu(\text{OH})$; $\nu(\text{C}=\text{O})$ and $\nu(\text{C}-\text{N})$ respectively.

In the spectrum of [Hg(Saln) $_2$] complex showed absence the $\nu(\text{OH})$ band, which showed in free ligand at (3302) cm $^{-1}$ suggested the deprotonated of hydroxyl group and bonded of Hg ion with hydroxate oxygen atom [16-20], and the $\nu(\text{C}=\text{O})$ was shifted to lower frequency from that of the free ligand, and showed at (1624)cm $^{-1}$, this shifted indicates that the carbonyl group was coordinated with Hg ion through the oxygen atom [19,20].

The spectra of complexes [2-9], showed the $\nu(\text{C}=\text{O})$ within (1636-1662) cm $^{-1}$ range, is higher than from the $\nu(\text{C}=\text{O})$ of complex (1) (1624) cm $^{-1}$, which suggest that the carbonyl group are non-involved in coordination to Hg ion [18-20], and suggesting a monodentate of (Saln $^-$) ligand through oxygen atom of the deprotonated hydroxyl group [16-20]. The spectra 2-9 displayed new bands at (1435-1450) cm $^{-1}$ and (498-513) cm $^{-1}$ range assigned to $\nu(\text{P}-\text{Ph})$ and $\nu(\text{P}-\text{C})$ [21-23], indicating the presence of the phosphine ligands in the complexes. This was supported by the appearance of a new medium intensity bands within the (463- 489)cm $^{-1}$ range in the spectra of the complexes which assigned to stretching frequency of $\nu(\text{Hg}-\text{P})$ bond [24-28]. Other selected bands are showed in **Table 2**.

Table 2 . Selected IR stretching vibration bands (cm $^{-1}$) of the prepared complexes (1-8)

Seq.	compounds	$\nu(\text{O}-\text{H})$	$\nu(\text{NH})$	$\nu(\text{C}-\text{H})$		$\nu(\text{C}=\text{O})$ ν	$\nu(\text{CN})$	$\nu(\text{Ph}-\text{P})$	$\nu(\text{P}-\text{C})$	$\nu(\text{P}-\text{Hg})$
				Ar.	Alph.					
1	HSaln	3148 _m	3302 _m	3059 _m	----	1618 _s	1558 _s	----	----	----
2	[Hg(Saln) $_2$]	----	3304 _m	3059 _m	----	1614 _s	1548 _s	----	----	----
3	[Hg(Saln) $_2$ (dppe)]	----	3308 _m	3055 _w	2920 _w	1662 _s	1548 _s	1437 _s	511 _m	478 _w
4	[Hg(Saln) $_2$ (dppp)]	----	3308 _m	3055 _w	2926 _w	1662 _s	1548 _s	1444 _m	509 _m	478 _w
5	[Hg(Saln) $_2$ (dppb)]	----	3310 _s	3055 _w	2928 _w	1647 _m	1548 _s	1440 _s	513 _m	482 _w
6	[Hg(Saln) $_2$ (dppf)]	----	3309 _w	3055 _w	----	1643 _s	1543 _s	1448 _m	498 _w	463 _w
7	[Hg(Saln) $_2$ (dppmS $_2$)]	----	3309 _m	3053 _w	2924 _w	1636 _s	1541 _s	1450 _m	507 _m	489 _w
8	[Hg(Saln) $_2$ (SPPH $_3$) $_2$]	----	3321 _w	3057 _w	----	1641 _s	1546 _s	1444 _s	513 _m	487 _s
9	[Hg(Saln) $_2$ (PPh $_3$) $_2$]	----	3304 _w	3053 _w	----	1658 _s	1541 _s	1435 _m	506 _m	483 _w

s: strong, m: medium, w : weak

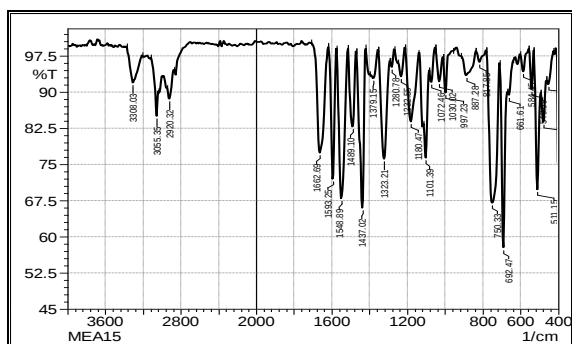


Fig. 1: IR spectrum of complex 2

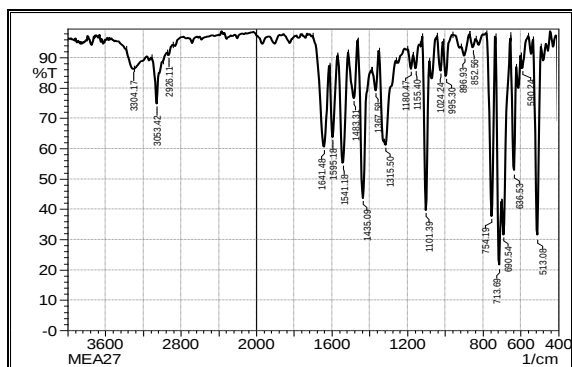
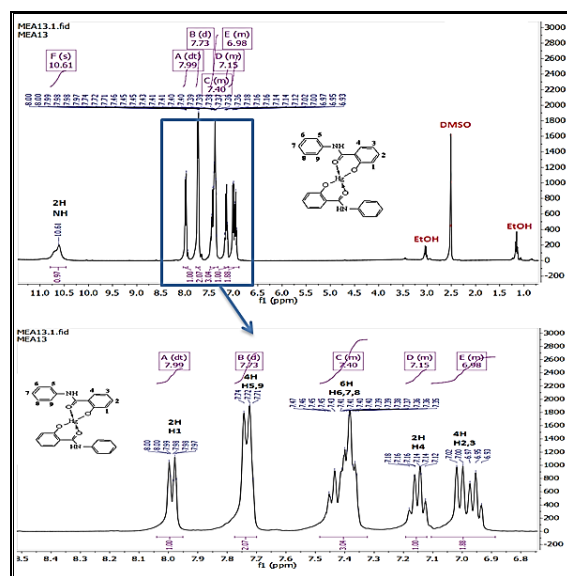


Fig. 2: IR spectrum of complex 7

3.3 NMR spectra

The ^{31}P and ^1H chemical shifts are listed in Table in 3. The ^1H NMR spectrum of complex (1) (Fig 3) showed six peaks, a singlet peak at $\delta 10.61$ ppm a sign to NH group, and two peaks at $\delta 7.99$ ppm and $\delta 7.73$ ppm with coupling constant ($^3J_{\text{HH}}$: 7.87 Hz and 8.00 Hz) due to the protons (H1) and (H5, 9) respectively. And three multiplets peaks

showed at $\delta 7.40$, $\delta 7.15$ and $\delta 6.98$ ppm refer to the protons (H6,7,8), (H4) and (H2,3) respectively.

Fig. 3: ^1H NMR spectrum of complex 1 in DMSO-d_6

The $^{31}\text{P}\{-^1\text{H}\}$ nmr spectra of the prepared complexes (2-7) (Fig 4 and 5) showed a single peak at δP = 30.33, 29.93, 30.37, 24.97, 34.10, and 42.29, ppm respectively, which indicates the presence of a single isomer each. The $^1\text{H}\{-^{31}\text{P}\}$ NMR of complexes (2-7) (Fig 6 and 7) showed the expected signals for the salicylanilide ligand as well as and the phosphine ligands.

Table 3 : ^1H and $^{31}\text{P}\{-^1\text{H}\}$ NMR chemical shifts (δ ppm) for the prepared complexes (1-7) measured in DMSO-d_6

Seq.	Complexes	δP (ppm)	δH^* (ppm)
1	$[\text{Hg}(\text{Saln})_2]$	----	10.61 (s, 2H, NH); 7.99 (d, $^3J_{\text{H-H}} = 7.87$ Hz, 2H, H1) ; 7.73 (d, $^3J_{\text{H-H}} = 7.87$ Hz, 4H, H5,9); 7.40 (m, 6H, H6,7,8); 7.15 (m, 2H, H4); 6.98 (m, 4H, H2,3).
2	$[\text{Hg}(\text{Saln})_2(\text{dppe})]$	30.33	10.35 (s, 2H, NH); 6.55 – 7.88 (m, 38H, H-Ar), 2.42 (s, 4H, CH_2 -dppe).
3	$[\text{Hg}(\text{Saln})_2(\text{dppp})]$	29.93	10.35 (s, 2H, NH); 6.70 – 7.92 (m, 38H, H-Ar), 3.07 (bs, 4H, CH_2 -dppp), 1.70 (s, 2H, CH_2 -dppp).
4	$[\text{Hg}(\text{Saln})_2(\text{dppb})]$	30.37	10.70 (s, 2H, NH); 6.91 – 8.02 (m, 38H, H-Ar), 2.36 (m, 4H, CH_2 -dppb), 1.57 (m, 4H, CH_2 -dppb).
5	$[\text{Hg}(\text{Saln})_2(\text{dppf})]$	24.97	10.35 (s, 2H, NH); 6.74 – 7.93 (m, 38H, H-Ar), 4.35 (bs, 4H, dppf), 4.59 (bs, 4H, dppf).
6	$[\text{Hg}(\text{Saln})_2(\text{dppmS}_2)]$	34.10	10.61 (s, 2H, NH); 6.99 – 8.01 (m, 38H, Ar-H), 4.59 (t, $^3J_{\text{P-H}} = 9.80$ Hz, 2H, CH_2 -dppmS ₂).
7	$[\text{Hg}(\text{Saln})_2(\text{SPPH}_3)_2]$	42.29	10.63 (s, 2H, NH); 6.93-7.99 (m, 48H, H-Ar),

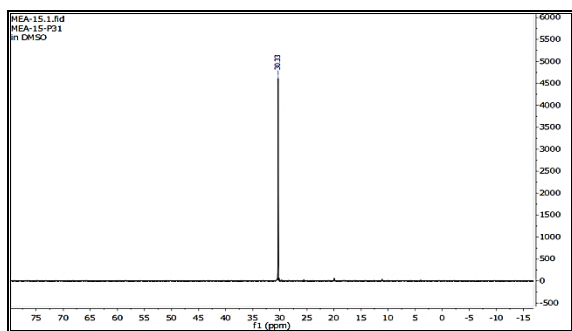


Fig. 4: $^{31}\text{P}\{-^1\text{H}\}$ nmr spectrum of complex (2) in DMSO-d^6

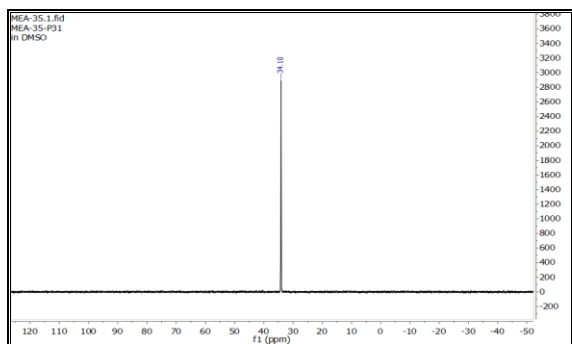


Fig. 5: $^{31}\text{P}\{-^1\text{H}\}$ nmr spectrum of complex (7) in DMSO-d^6

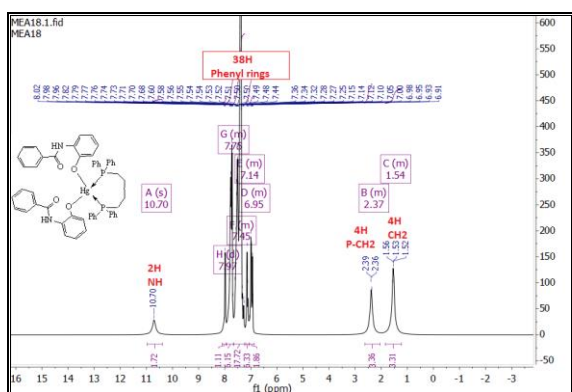


Fig. 6: $^1\text{H}\{-^{31}\text{P}\}$ nmr spectrum of complex (4) in DMSO-d^6

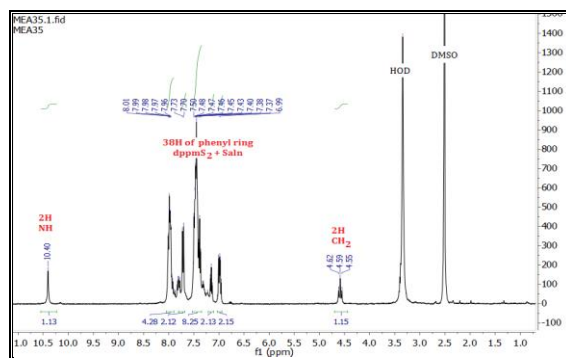


Fig. 7: ^1H nmr spectrum of complex (7) in DMSO-d^6

4. Conclusions

In summary, we have synthesized an eight new salicylanilide Hg(II) complexes by the reaction of the (HSal⁻) ligand with mercury acetate to afford complex of the type $[\text{Hg}(\kappa^2\text{-Saln})_2]$. A complexes of the types $[\text{Hg}(\kappa^1\text{-Saln})_2(\text{diphos})]$ (2-6) or $[\text{Hg}(\kappa^1\text{-Saln})_2(\text{phos})_2]$ (7,8) were prepared by reaction of complex (1) with diphosphine or monophosphine respectively. The prepared complexes have been characterized by CHN elemental analyses, molar conductivity, IR and NMR (^1H , and ^{31}P) spectroscopy. In complex (1) the (Saln⁻) was bonded as bidentate chelating ligand through the oxygen atoms of the carbonyl and deprotonated hydroxyl group, whereas bonded as monodentate ligand through the oxygen atom of deprotonated hydroxyl group in all other complexes (2-8). The geometry of the complexes (1-8) is a tetrahedral around the mercury (II) ion.

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معقدات جديدة لليكاند الساليسيلانيليد - الزئبق (II) مع الفوسفينات، تحضير وتشخيص طيفي

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الملخص

خُضر المعقد الجديد ذي الصيغة $[Hg(\kappa^2\text{-Saln})_2]$ (1) من تفاعل خلات الزئبق مع ليكاند الساليسيلانيليد في الايثانول بنسبة مولية (2:1) وبوجود ثلاثي اثيل امين كقاعدة. ثم تم معاملة المعقد الناتج مع مول واحد من الفوسفينات الثنائية dppf, dppb, dppp, dppe, dppm لينتج معقدات ذات صيغة $[Hg(\kappa^1\text{-Saln})_2(\text{diphos})]$ (2-6)، او مع مولين من ثلاثي فينيل فوسفين سلفايد $SPPH_3$ او ثلاثي فينيل فوسفين PPh_3 لينتج معقدات ذات صيغة $[Hg(\kappa^1\text{-Saln})_2(\text{phos})_2]$ (7 و 8). شُخصت المعقدات المحضرة بواسطة التحليل الدقيق للعناصر، الموصلية المولارية الكهربائية، مطيافية الرنين النووي المغناطيسي للبروتون، الفسفور ومطيافية الاشعة تحت الحمراء، وقد وجد ان ليكاند الساليسيلانيليد في المعقد (1) يسلك سلوك ليكاند ثنائي السن عن طريق ذرة الاوكسجين لمجموعة الكاربونيل واوكسجين مجموعة الهيدروكسيل بعد فقدان البروتون، في حين يسلك كليكاند احادي السن عن طريق ذرة اوكسجين مجموعة الهيدروكسيل الفاقد للبروتون في المعقدات (2-8)، لتنتج شكلاً رباعي السطوح حول ايون الزئبق (II).