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Preparation and crystallographic characterization of Pd(II) complexes containing imidobis(diphenylthiophosphinato) ligand

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Abstract

The reactions of $PdCI_2(L-L)$ [L-L = $Ph_2PCH_2PPh_2(dppm)$, $Ph_2PCH_2CH_2PPh_2(dppe)$ and $Ph_2PCH_2CH_2CH_2PPh_2(dppp)$] with equivalent amount of $(Ph_2P(S)NHP(S)Ph_2)(dppaS_2)$ gave the complexes $[Pd(L-L)(dppaS_2-H)]ClO_4$ [L-L = dppm (1), dppe (2), dppp (3)]. The different synthetic route was used for complex 2 by using of $Pd(dppe)Cl_2$ and $K[N(PSPh_2)_2]$ as starting materials (2a). All of these complexes have been characterized ${}^{31}P{}^{1}H{}$ NMR, IR and elemental analyses. The complexes 2, 2a and 3 were crystallographically characterized. The coordination geometry around the Pd atoms in these complexes distorted square planar. Six membered dppaS_2-H rings are twist boat conformations in three complexes.

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1. Introduction

Ligands with mixed donor sets have attracted much interesting especially in recent years [1]. One particular class that has received widespread attention is hemilabile ligands in which both "soft" (e.g. P) and "hard" (e.g. N or O) donor centres are present [2,3]. The hard donor site coordinates only weakly to a late transition-metal centre and can readily be displaced by other ligands. This property has been extensively exploited in catalysis [4]. The iminobis(diphenylphosphinechalcogenide) ligands may be considered to be main group analogues of acetylacetone [5]. Dichalcogenoimidodiphosphinato anions $[R_2P(E)-N-P(E)R_2]^-$ (E = O, S, Se), derived from iminobis(diphenylphosphinechalcogenide) ligands, with a strong tendency to from inorganic (carbon-free) chelate rings. All dichalcogenoimidodisphosphinates 1 tend to

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form six-membered inorganic (carbon-free) chelate rings 2 and seldom act as bridging ligands [6].

With differing donor properties of the atoms (O, S or Se) at the two ends of the diphosphazene fragment, the comparison of the structures and properties of various dichalcogenoimidodiphosphinato ligand becomes interesting [6]. Gabor Balazs et al. have reported the reaction between Li[HN(X)PR₂] and $R'_2P(Y)Cl$ in a benzene/*n*-hexane mixture to give white crystalline solids of (XPPh₂)-[YP(OEt)₂]NH (X, Y = O, S) [7].

As a result a number of complexes have been described. The reaction of $Cu(PPh)_3NO_3$ with $K[Ph_2P(Se)-N-P(Se)Ph_2]$ has been reported by Novasad [6]. Martin B. Smith has reported a number of Pd(II) and Pt(II) complexes with the ligands $Ph_2PNHP(O)Ph_2$, $[Ph_2PNP(O)-Ph_2]^-$ or $[Ph_2P(E)NP(O)Ph_2]^-$ (E = S or Se) [8]. It has been reported heterofunctional ligands of the type $R_2PNHP(O)$ - R_2 [9,10] and $[R_2P(E)NP(O)R_2]^-$ (E = S or Se; R = Ph) [11–13] and their coordination chemistry to catalytically useful metals. Manganese compounds of the type $Mn[(XPR_2)(YPR'_2)N]_2(X, Y = O, S; R, R' = Me, Ph)$ were

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prepared by metathesis reactions between $MnCl_2 \cdot 4H_2O$ and the alkaline salt of the corresponding ligand by Szekely et al. [14].



In this study, we are reporting the preparation of cationic palladium(II) complexes containing $[Ph_2P(S)-NP(S)Ph_2]^-$ [disulfideimidodiphosphinato] of the type $[Pd(L-L)(dppaS_2-H)]ClO_4 [L-L = dppm (1), dppe (2), dppp (3)]$. Complex 2 was also synthesized 2a by using of Pd(dppe)Cl₂ and K[N(PSPh₂)₂] as starting materials. All of these complexes have been characterized ³¹P{¹H} NMR, IR and elemental analyses. Complex 2 was recrystallized from acetone/hexane, complex 3 was recrystallized from dichloromethane/diethyl ether for X-ray crystallography.

2. Results and discussion

Previous works have demonstrated that the iminobis(phosphinechalcogenide) ligands can be deprotonated to afford coordinated dichalcogenoimidodiphosphinato anions by the reaction of metal-halides or starting complexes containing metal-halide bonds [15]. The starting complexes, $Pd(L-L)Cl_2$ (L-L = dppm, dppe, dppp) and $K[N(PP_2S)_2]$ were synthesized according to the literature (see Section 2). ${}^{31}P{}^{1}H{}$ NMR spectra of these compounds only a singlet δP values are -51.17, 67.67, 13.64 and 36.70 ppm, respectively. [Pd(L-L)(dppaS₂-H)]ClO₄ complexes (1, 2, 3) were prepared by the mixing of solution of Pd(L-L)Cl₂ complexes in CH₂Cl₂ and dppaS₂ ligand and NaClO₄ \cdot H₂O salt in (CH₃)₂CO. Another method was used to prepared [Pd(dppe)(dppaS₂-H)]ClO₄ (2a). In this method, the solution of $K[N(PP_2S)_2]$ salt in CH₃OH was added solution of Pd(dppe)Cl₂ in CH₂Cl₂ as a molar ratio 1:1 in the presence of NaClO₄ \cdot H₂O salt.

Solid state IR spectra of the complexes (1–3) showed the characteristic absorption of P=S group at 570.0, 573.0 and 570.0 cm⁻¹. These absorptions are shifted to the lower frequencies according to the free ligand, dppaS₂, absorption which is at 612.9 cm⁻¹. This shows that the ligand is coordinating to Pd(II) center via S donor atoms in the complexes. ClO_4^- group vibrations give rise to two bonds in 1100 and 620 cm⁻¹ region. IR spectra of free acid, dppaS₂, exhibits absorption of medium intensity at 2630.8 cm⁻¹ and very strong absorption at 922.0 cm⁻¹ which were assigned to v(N-H) stretching vibration and $v_{as}(P_2NH)$ stretching vibration, respectively. In IR spectra of complexes **1**, **2**, **3**,

presence of strong absorptions in the regions 1150– 1170 cm⁻¹, assigned to the $v_{as}(P_2NH)$ stretching vibrations, indicate that the dppaS₂ ligand is coordinated to the palladium center in the deprotonated form [6]. In K[N(PP₂S)₂] salt, which is prepared to obtain [Pd(dppe)(dppaS₂-H)]CIO₄ **2a** by different method, $v_{as}(P_2N)$ vibration is at 1171.2 cm⁻¹. This absorption is at 1146.7 cm⁻¹ in complex **2a**. Analytical data of four complexes are in accordance with the proposed formulation.

In complexes 1, 2, 3 and 2a, the resonance due to the diphosphines (dppm, dppe, dppp) phosphorus atoms appear at -42.25, 72.06, 11.35 and 69.55 ppm, respectively. It has been seen that the signals of chelate dppm and dppe ligands are shifted to the downfield related to the starting complexes $[Pd(L-L)Cl_2]$ (L-L = dppm, dppe), but dppp ligand is shifted to the upfield on relative to the starting complex, Pd(dppp)Cl_2. Phosphorus chemical shifts of dppaS₂-H are 36.16, 38.67 and 37.63 ppm in complexes 1, 2 and 3, respectively.

In ${}^{31}P{}^{1}H$ NMR of pottassium salt, K[N(PP₂S)₂], there is a shifted at 38.69 ppm. This resonance is at 38.65 ppm in complex **2a** and is suitable with value of the complex **2**.

Solid state structures of complexes 2, 2a, and 3 were determined by single crystal X-ray diffraction. Complex 2 was recrystallized from acetone/hexane and includes one molecule acetone, complex 2a was recrystallized from $CH_2Cl_2/$ diethyl ether and has one molecule CH₂Cl₂. Complex 3 was recrystallized from acetone/diethyl ether and includes one molecule acetone. The perspective view of complexes 2, 2a and 3 are given in Figs. 1–3, respectively. The selected bond distances and angles are listed in Table. 1. Complexes 2 and 2a are monoclinic and complex 3 is triclinic crystal systems. The solvent (CH₃)₂CO and CH₂Cl₂ lie in the lattice on non-bonded molecules in the complexes 2 and 3, respectively. All of the data for complex 2 and 2a are similar. Palladium atoms in every complex are distorted square planar environments with from P(3)Pd(1)S(1) and P(4)Pd(1)S(2)are 168.90(4)° and 169.99(4)° for complex 2, 170.66(3)° and 170.92(3)° for complex 2a and 171.57(2)° and 173.96(2)° for complex 3. The bite angles of iminobis(diphenylphosphine sulfide) in complexes 2 and 2a [102.94(3)° and $102.69(3)^{\circ}$) are different from complex 3 (98.82(2)°]. This difference can be explained by diphosphine bite angle $[P(4)Pd(1)P(3); 84.85(4)^{\circ} \text{ for complex } 2 \text{ and } 84.61(3)^{\circ} \text{ for}$ complex 2a and $91.62(2)^{\circ}$ for complex 3]. These values are comparable with the complexes $[Pd(dppaS_2-H)_2][5]$.

Pd–S bond lengths are slightly different from each other. Pd(1)–S(1) and Pd(1)–S(2) are 2.3933(10) and 2.4114(10) Å for complex **2**; 2.3853(9) and 2.4110(9) Å for complex **2a**; 2.4017(6) and 2.3674(6) Å for complex **3**. These are comparable with the literature values [16].

In complex 3, six membered dppp chelate ring has chair conformation with Pd(1)P(3)C(13)C(14) and Pd(1)P(4)-C(15)C(14) torsion angles of $-44.4(3)^{\circ}$ and $60.2(2)^{\circ}$, respectively, and with Pd(1) and C(14) atoms are located at -0.6932(21) Å below and 0.7342(45) Å above the plane defined by the atoms P(3)-P(4)-C(13)-C(15).



Fig. 1. The perspective view of **2** (20% thermal probability). Hydrogen atoms, anions and solvent molecule are omitted for clarity.



Fig. 2. The perspective view of **2a** (20% thermal probability). Hydrogen atoms, anions and solvent molecule are omitted for clarity.

Six membered dppaS₂-H rings are twist boat conformations in three complexes and are very similar to that observed related platinum analogue $[Pt(PEt_3)_2\{Ph_2P(S)-NHP(S)Ph_2\}]PF_6$ in the literature [17].

PNP bond angles are $122.41(19)^\circ$, $120.74(17)^\circ$ and $121.47(13)^\circ$, respectively. These angles show the sp² character at the nitrogen atoms [5].

P–S bond lengths are 2.0184(14) and 2.0170(15) Å for complex **2**; 2.0165(11) and 2.0239(12) Å for complex **2a** and 2.0304(9) and 2.0152(9) Å for complex **3**. P–S bond distances, as expected, are longer than the analogous P=S distances in the sulfur compounds and are suitable with the similar compounds in the literature [6,7,14].



Fig. 3. The perspective view of 3 (20% thermal probability). Hydrogen atoms, anions and solvent molecule are omitted for clarity.

Table 1

Selected bond angles (°) and distances (Å) for $[Pd(dppe)(dppaS_2-H)]-(ClO_4) \cdot (CH_3)_2CO$ (2), $[Pd(dppe)(dppaS_2-H)](ClO_4) \cdot CH_2Cl_2$ (2a) and $[Pd(dppp)(dppaS_2-H)](ClO_4) \cdot (CH_3)_2CO$ (3)

	2	2a	3
Bond distances			
Pd(1) - S(1)	2.3933(10)	2.3853(9)	2.4017(6)
Pd(1)–S(2)	2.4114(10)	2.4110(9)	2.3674(6)
P(1) - S(1)	2.0184(14)	2.0165(11)	2.0304(9)
P(2)–S(2)	2.0170(15)	2.0239(12)	2.0152(9)
Pd(1) - P(3)	2.2667(10)	2.2616(9)	2.2915(6)
Pd(1) - P(4)	2.2622(11)	2.2614(9)	2.2819(6)
Bond angles			
S(1) - Pd(1) - S(2)	102.69(3)	102.94(3)	98.82(2)
P(4)-Pd(1)-S(2)	169.99(4)	170.92(3)	173.96(2)
P(3) - Pd(1) - P(4)	84.85(4)	84.61(3)	91.62(2)
P(3)-Pd(1)-S(1)	168.90(4)	170.66(3)	171.57(2)
P(3)-Pd(1)-S(2)	88.13(3)	86.39(3)	84.50(2)
P(4)-Pd(1)-S(1)	84.72(4)	86.06(3)	85.66(2)

As a result, three new complexes have been synthesized. Two different synthetic route was used for one of them and their structures were determined by spectroscopic methods.

3. Experimental

Solvents were dried according to the methods given in the literature and were purified under inert conditions [18]. All synthesis were carried using standard Schlenk tube techniques under inert atmosphere.

dppm, dppe, dppp, NaClO₄ · H₂O and acetone were purchased from Merck Chemical; dichloromethane, toluene, methanol, hexane and diethylether were purchased from Lab-Scan. The starting complexes, $[PdCl_2(COD)]$ (COD = 1,5-cyclooctadiene) [19], which is used for preparing the $[PdCI_2(L-L)]$ complexes $[L-L = dppm (Ph_2PCH_2-PPh_2)$, dppe $(Ph_2PCH_2CH_2PPh_2)$, dppp $(Ph_2PCH_2CH_2-CH_2PPh_2)]$ [20], dppa $(Ph_2PNHPPh_2)$ and dppaS₂ $(Ph_2P(S)NHP(S)Ph_2)$ were synthesized according to the method given in the literature [21].

 ${}^{31}P{}^{1}H$ NMR spectra were recorded on Varian AS 400+Mercury instruments, chemical shifts, in ppm, related to external 85% H₃PO₄. Coupling constants are in Hz. IR spectra were recorded from 4000 to 400 cm⁻¹ with a Per-kin–Elmer 100 FT-IR instrument. Melting points were determined by Electrothermal melting point detection apparatus. Elemental analyses were performed by the TÜBİTAK-Ankara Test and Analyse Laboratories. There is potential explosion hazard arising from the use of perchlorates in the presence of organic material, so quantities should be kept small and heating (especially to dryness) avoided.

3.1. Method I

3.1.1. $[Pd(dppm)(dppaS_2-H)](ClO_4)$ (1)

PdCl₂(dppm) (0.18 mmol, 0.10 g) and dppaS₂ (0.18 mmol, 0.08 g) were placed in 30 mL CH₂Cl₂and stirred. The solution of NaClO₄ \cdot H₂O (0.71 mmol, 0.10 g) in 20 mL acetone was added dropwise to the stirring solution. Stirring was continued for 24 h at room temperature. After that, the solution was removed in vacuo. Yellow solid was dissolved in CH₂Cl₂ and then it was filtered through celite in order to separate NaCI. The solution was removed in vacuo again. Complex 1 was recrystallized from acetone/

diethyl ether (1:3 v/v) to give yellow crystals. Yield 0.08 g, 41.9%. Data for 1: ${}^{31}P{}^{1}H{}$ NMR (DMSO): -42.30 ppm (s), 36.18 ppm (s). Selected IR data (KBr): v(PS) 570.0 cm⁻¹, 1098.5 (s) and 622.7 (m) cm⁻¹ (ClO₄⁻¹). *Anal.* Calc. for C₄₉H₄₂NClO₄P₄PdS₂: C, 56.65; H, 4.04; S, 6.17; N, 1.34. Found: C, 56.12; H, 4.16; S, 5.80; N, 1.65%.

3.1.2. $[Pd(dppe)(dppaS_2-H)](ClO_4)_2$ (2)

[PdCl₂(dppe)] (0.17 mmol, 0.10 g), dppaS₂ (0.17 mmol, 0.08 g) were placed together in 30 mL CH₂CI₂ and stirred. The solution of NaClO₄ · H₂O (0.71 mmol, 0.10 g) in 20 mL acetone was added dropwise to the stirring solution. After 24 h, the solution was removed in vacuo. The solid was dissolved in CH₂CI₂ and then it was filtered through celite to separate NaCl. After the resulting solution was removed in vacuo this complex was obtained as a cream solid and it was recrystallized from acetone/hexane. (1:3 v/v). Yield 0.07 g, 40%. Data for **2**: ³¹P{¹H} NMR (DMSO): 72.13 ppm (s), 38.76 ppm (s). Selected IR data (KBr): v(PS) 573.0 cm⁻¹, 1098.5 (s) and 622.7 (m) cm⁻¹ (ClO₄⁻). *Anal.* Calc. for C₅₀H₄₄NClO₄P₄PdS₂: C, 57.04; H, 4.21; S, 6.09; N, 1.33. Found: C, 56.44; H, 4.15; S, 5.53; N, 1.76%.

3.1.3. $[Pd(dppp)(dppaS_2-H)](ClO_4)_2$ (3)

This complex was obtained as a yellow solid, in a manner similar to **1** and **2** from $[PdCl_2(dppp)]$ (0.17 mmol, 0.10 g), dppaS₂ (0.17 mmol, 0.08 g) and NaClO₄ · H₂O (0.71 mmol, 0.10 g). Yellow crystals, suitable for X-ray analysis, were obtained by slow diffusion

Table 2

Crystal data, data collection and structure-refinement parameters for complex 2, 2a and 3

Empirical formula	2	2a	3
	$\overline{C_{50}H_{44}NClO_4P_4PdS_2\cdot(CH_3)_2CO}$	$C_{50}H_{44}NClO_4P_4PdS_2\cdot CH_2Cl_2$	$C_{51}H_{46}NClO_4P_4PdS_2\cdot(CH_3)_2CO$
Color	light yellow	colorless	yellow
Crystal size (mm)	$0.750 \times 0.333 \times 0.070$	$0.560 \times 0.353 \times 0.190$	$0.640 \times 0.440 \times 0.330$
Crystal system	monoclinic	monoclinic	triclinic
Space group	P21/c	P21/c	$P\overline{1}$
Unit cell dimensions			
a (Å)	17.2474(8)	13.2643(6)	11.0367(5)
b (Å)	11.1817(4)	27.6872(8)	13.1618(6)
<i>c</i> (Å)	27.3800(12)	15.3404(7)	20.6186(9)
α (°)	90.00	90.00	73.135(3)
β (°)	98.217(4)	111.568(3)	77.898(4)
γ (°)	90.00	90.00	68.038(3)
Volume (Å ³)	5226.2(4)	5239.3(4)	2641.0(2)
Ζ	4	4	2
$D_{\rm calc} ({ m Mg}{ m m}^{-3})$	1.412	1.442	1.414
Formula weight	1110.79	1137.64	1124.82
Absorption coefficient (mm ⁻¹)	0.656	0.753	0.650
F(000)	2280	2320	1156
Index ranges	$-20 \leqslant h \leqslant 20,$	$-15 \leqslant h \leqslant 15$,	$-12 \leqslant h \leqslant 13$,
	$-13 \leqslant k \leqslant 13$,	$-32 \leqslant k \leqslant 32$,	$-16 \leqslant k \leqslant 16$,
	$-32 \leqslant l \leqslant 32$	$-18 \leqslant l \leqslant 18$	$-25 \leqslant l \leqslant 25$
θ Range (°)	1.50-25.03	1.47-25.04	1.72-26.00
Number of data measured	9164	9190	10375
Number of data with $[I \ge 2\sigma(I)]$	5612	7266	8827
$R_1 \left[I \ge 2\sigma(I) \right]$	0.0413	0.0397	0.0339
$WR_2 [I > 2\sigma(I)]$	0.0714	0.1117	0.0933

of diethylether into a acetone solution of **3**. (1:3 v/v). Yield 0.09 g, 50.5%. Data for **3**: ${}^{31}P{}^{1}H{}$ NMR (DMSO): 11.39 ppm (s), 37.67 ppm (s). Selected IR data (KBr): v(PS) 570.0 cm⁻¹, 1098.5 (s) and 622.7 (m) cm⁻¹ (ClO₄⁻¹). *Anal.* Calc. for C₅₁H₄₆NClO₄P₄PdS₂: C, 57.42; H, 4.31 ; S, 6.01; N, 1.31. Found: C, 56.25; H, 4.41; S, 5.20; N, 1.50%.

3.2. Method II

3.2.1. $[Pd(dppe)(dppaS_2-H)](ClO_4)_2$ (2a)

dppaS₂ (0.45 mmol, 0.2 g) and KOBu^t (0.45 mmol, 0.05 g) were stirred in diethyl ether and K[N(SPPh₂)₂] synthesized.

K[N(SPPh₂)₂] (0.17 mmol, 0.085 g) in 5 mL methanol and NaClO₄ · H₂O (0.71 mmol, 0.10 g) in 2 mL methanol was added to PdCl₂dppe (0.17 mmol, 0.1 g) in 15 mL CH₂Cl₂. After 24 h, the solvent was removed in vacuo. Complex **2a** was recrystallized from CH₂Cl₂/diethyl ether (1:3 v/v). Yield 0.11 g, 58.08%. Data for **2a**: ³¹P{¹H} NMR (CDCl₃): 69.5 ppm (s), 38.6 ppm (s). Selected IR data (KBr): v(PS) 576.0 cm⁻¹, 1093.7 (s) and 623.1 (m) cm⁻¹ (ClO₄⁻). *Anal.* Calc. for C₅₀H₄₄NClO₄P₄PdS₂: C, 57.04; H, 4.21; S, 6.09; N, 1.33. Found: C, 56.09; H, 4.38; S, 5.5; N, 1.65%.

3.2.2. X-ray crystallographic study of 2, 2a and 3

The intensity data of the complexes 2, 2a and 3 were collected using a STOE IPDS 2 diffractometer with graphitemonochromated Mo K α radiation ($\lambda = 0.71073$) at 296 K. Details of crystal data, data collection, structure solution and refinement are given in Table 2. Data collection: Stoe X-AREA [22]. Cell refinement: Stoe X-AREA [22]. Data reduction: Stoe X-RED [22]. The structure was solved by direct-methods using SHELXS-97 [23] and anisotropic displacement parameters were applied to non-hydrogen atoms in a full-matrix least-squares refinement based on F^2 using SHELXL-97 [24]. All hydrogen atoms were included using a riding model. Molecular drawings were obtained using ORTEP-III [25].

4. Supplementary material

CCDC 622361, 622362 and 622363 contains the supplementary crystallographic data for $C_{50}H_{44}NClO_4$ - $P_4PdS_2 \cdot (CH_3)_2CO$, $C_{50}H_{44}NClO_4P_4PdS_2 \cdot CH_2Cl_2$ and $C_{51}H_{46}NClO_4P_4PdS_2 \cdot (CH_3)_2CO$ respectively. These data can be obtained free of charge via http://www.ccdc.ac.uk/conts/retrieving.html, or from the Cambridge Crystallo-

graphic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

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