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Monodentate *S*-*vs*. bidentate 1,5-*O*,*S*-coordination of *N*-phosphoryl-*N*′-(R)-thioureas with Pd(II)

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ABSTRACT

Reaction of *N*-phosphorylated thioureas of common formula RNHC(S)NHP(O)(O*i*Pr)$_2$ (R = *t*Bu, **HL**^{I}; R = Ph, **HL**^{II}; R = 4′-benzo-15-crown-5, **HL**^{III}) with Pd(PhCN)$_2$Cl$_2$ in acetonitrile leads to complexes of the structure Pd(**HL**^{I}-*S*)$_2$Cl$_2$ (**1**), Pd(**HL**^{II}-*S*)$_2$Cl$_2$ (**2**) and Pd(**HL**^{III}-*S*)$_2$Cl$_2$ (**3**). Reaction of *N*-phosphorylated thioureas of common formula RC(S)NHP(O)(O*i*Pr)$_2$ (R = Et$_2$N, **HL**^{IV}; R = morpholine-*N*-yl, **HL**^{V}) in the same conditions leads to complexes Pd(**L**^{IV}-*O*,*S*)$_2$ (**4**) and Pd(**L**^{V}-*O*,*S*)$_2$ (**5**), where the palladium(II) atoms are coordinated in a square-planar fashion by the C═S sulfur atoms and the P═O oxygen atoms of two anionic ligands. The crystal structure of complex **1** has been investigated by X-ray crystallography. It was established that the thiourea ligands are in a *trans*-configuration and the palladium(II) cation is coordinated by the sulfur atoms of the C═S groups and the chlorine atoms. Complex **1** is the first example of palladium(II) complex in which the potentially chelating *N*-phosphorylated thiourea ligand is bound through the sulfur atom only.

1. Introduction

The coordination chemistry of polyfunctional ligands, capable to realize different coordination modes with metal cations [1] is of interest for synthesis of new selective complexing agents and analytical reagents. Koch and others have shown that the coordination of *N*,*N*-dialkyl-*N*′-aroylthioureas leads to bidentate-*O*,*S*-coordination to Pd(II) [2,3]. It was also shown that the coordination mode of *N*-alkyl-*N*′-aroylthioureas depends on an intramolecular hydrogen bond between the donor oxygen atom of the carbonyl group and the hydrogen atom of the NH group [2].

We have become interested in the coordination chemistry of *N*-phosphorylated thioureas RR′NC(S)NHP(O)(O*i*Pr)$_2$ (R = R′ = alkyl; R = H, R′ = alkyl, aryl) towards various transition metal cations [4] in view of their application in metals separation [5]. Recently, we reported, that in the square-planar complexes of *N*-phosphorylthioureas M[RNHC(S)NP(O)(O*i*Pr)$_2$]$_2$, where (M = Ni(II), R = Ph [6], *p*-MeOC$_6$H$_4$, *p*-BrC$_6$H$_4$, *t*Bu, *i*Pr, *c*-Hex [7]; M = Pd(II), R = Ph [6], *i*Pr [7]; M = Cu(II), R = Ph, *c*-Hex [6]), 1,3-*N*,*S*-coordination of the anionic ligand is realized.

Recently, we reported some synthetic results presented here as a preliminary communication [8]. Herein, we report the comparative study on the complexes of the Pd(II) ions with *N*-phosphorylthioureas, containing secondary (Scheme 1) and tertiary (Scheme 2) nitrogen at the *N*-thioacylamidophosphate moiety C(S)NHP(O)(O*i*Pr)$_2$. The crystal structure of *trans*-Pd(HL^{I}-*S*)$_2$Cl$_2$, the first example of *N*-phosphorylthiourea coordinated to Pd(II) only through the S atom in the monodentate fashion, is also reported.

2. Experimental

2.1. Synthesis of HL^{I}–HL^{V}

N-Diisopropoxyphosphorylthioureas were prepared according to the previously described methods [9].

2.2. Synthesis of **1**–**5**

A portion of Pd(PhCN)$_2$Cl$_2$ (0.192 g, 0.5 mmol) dissolved in 30 mL of acetonitrile was added dropwise to 30 mL of a well stirred solution containing **HL**$^{I-V}$ (0.296, 0.316, 0.506, 0.295 or 0.310 g; 1 mmol) in the same solvent. The mixture was stirred at room temperature for a further 10 h. The solvent was then removed *in vacuo*. A precipitate was isolated from dichloromethane by *n*-hexane.

2.2.1. Pd(HL^{I}–S)$_2$Cl$_2$ (**1**)

Yield: 0.331 g (86%). M.p. 128–129 °C. ^{1}H NMR (CDCl$_3$): δ 1.38–1.43 (m, 12H, CH$_3$), 1.49–1.53 (m, 9H, CH$_3$, *t*Bu), 4.58–4.73 (m, 2H,

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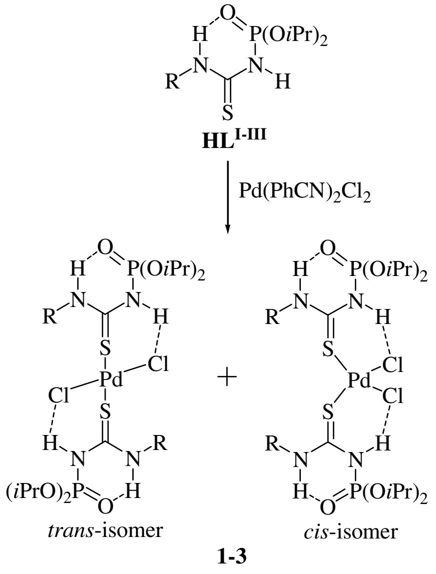
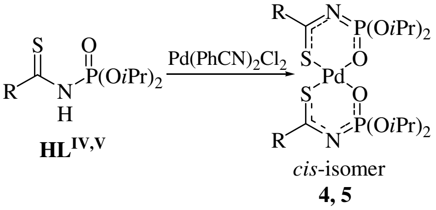


Scheme 1. Preparation of complexes **1–3** (R = *t*Bu, **HL**^{I} (**1**); Ph, **HL**^{II} (**2**); 4′-benzo-15-crown-5, **HL**^{III} (**3**)).



Scheme 2. Preparation of complexes **4** and **5** (R = Et_2N, **HL**^{IV} (**4**); morpholine-*N*-yl, **HL**^{V} (**5**)).

OCH), 9.61, 10.02 (2d, $^{2}J_{PNH}$ = 5.5 Hz, 1H, P(O)NH), 10.37, 10.49 (2s, 1 H, ArNH). $^{31}P\{^{1}H\}$ NMR ($CDCl_3$): δ −6.5, −6.8. IR: ν 1002 (POC), 1246 (P=O), 1534 (S=C–N), 3082, 3203 (NH) cm^{-1}. *Anal.* Calc. $C_{22}H_{50}Cl_2N_4O_6P_2PdS_2$ (770.06): C, 34.31; H, 6.54; N, 7.28. Found: C, 34.49; H, 6.62; N, 7.37%.

2.2.2. *Pd(HL^{II}–S)$_2$Cl$_2$ (**2**)*

Yield: 0.299 g (74%). M.p. 147–148 °C. ^{1}H NMR ($CDCl_3$): δ 1.41–1.48 (m, 12H, CH_3), 4.71–4.90 (m, 2H, OCH), 7.23–7.68 (m, 5H, C_6H_5), 9.80, 10.13 (2d, $^{2}J_{PNH}$ = 5.6 Hz, 1H, P(O)NH), 10.67, 10.79 (2s, 1H, PhNH). $^{31}P\{^{1}H\}$ NMR ($CDCl_3$): δ −5.6, −5.9. IR: ν 998 (POC), 1258 (P=O), 1521 (S=C–N), 3091, 3217 (NH) cm^{-1}. *Anal.* Calc. for $C_{26}H_{42}Cl_2N_4O_6P_2PdS_2$ (810.04): C, 38.55; H, 5.23; N, 6.92. Found: C, 38.32; H, 5.32; N, 6.78%.

2.2.3. *Pd(HL^{III}–S)$_2$Cl$_2$ (**3**)*

Yield: 0.483 g (81%). M.p. 139–141 °C. ^{1}H NMR ($CDCl_3$): δ 1.43–1.47 (m, 12H, CH_3), 3.65–4.21 (m, 16H, CH_2), 4.69–4.89 (m, 2H, OCH), 6.75–6.97 (m, 3H, C_6H_3), 9.88, 10.16 (2d, $^{2}J_{PNH}$ = 5.8 Hz, 1H, P(O)NH), 10.72, 10.83 (2s, 1H, ArNH). $^{31}P\{^{1}H\}$ NMR ($CDCl_3$): δ −6.0, −6.4. IR: ν 1008 (POC), 1140 (COC), 1241 (P=O), 1515, 1549 (S=C–N), 3094, 3211 (NH) cm^{-1}. *Anal.* Calc. for $C_{42}H_{72}Cl_2N_4O_{16}P_2PdS_2$ (1192.44): C, 42.30; H, 6.09; N, 4.70. Found: C, 42.72; H, 5.99; N, 4.59%.

2.2.4. *Pd(L^{IV}–O,S)$_2$ (**4**)*

Yield: 0.177 g (51%). M.p. 118–119 °C. ^{1}H NMR ($CDCl_3$): δ 1.03–1.26 (m, 12H, CH_3, Et_2N), 1.29–1.39 (m, 24H, CH_3, *i*PrO), 3.35–3.79 (m, 8H, CH_2, Et_2N), 4.57–4.74 (m, 4H, OCH). $^{31}P\{^{1}H\}$ NMR ($CDCl_3$): δ 9.4. IR: ν 1003 (POC), 1142 (P=O), 1529 (SCN) cm^{-1}. *Anal.* Calc. for $C_{22}H_{48}N_4O_6P_2PdS_2$ (696.15): C, 37.90; H, 6.94; N, 8.04. Found: C, 37.84; H, 6.99; N, 8.11%.

2.2.5. *Pd(L^{V}–O,S)$_2$ (**5**)*

Yield: 0.199 g (55%). M.p. 157–158 °C. ^{1}H NMR ($CDCl_3$): δ 1.33 (d, $^{3}J_{H,H}$ = 6.1 Hz, 12H, CH_3), 1.34 (d, $^{3}J_{H,H}$ = 6.1 Hz, 12H, CH_3), 3.62–3.97 (m, 16H, CH_2), 4.68 (d sept, $^{3}J_{P,H}$ = 7.6 Hz, $^{3}J_{H,H}$ = 6.1 Hz, 4H, OCH). $^{31}P\{^{1}H\}$ NMR ($CDCl_3$): δ 9.9. IR: ν 1005 (POC), 1144 (COC), 1154 (P=O), 1547 (SCN) cm^{-1}. *Anal.* Calc. for $C_{22}H_{44}N_4O_8P_2PdS_2$ (724.11): C, 36.44; H, 6.12; N, 7.73. Found: C, 36.49; H, 6.07; N, 7.68%.

2.3. *Physical measurements*

Infrared spectra (Nujol) were recorded with a Specord M-80 spectrometer in the range 400–3600 cm^{-1}. NMR spectra were obtained on a Varian Unity-300 NMR spectrometer at 25 °C. ^{1}H and $^{31}P\{^{1}H\}$ spectra were recorded at 299.948 and 75.429 MHz, respectively. Chemical shifts are reported with reference to $SiMe_4$ (^{1}H) and H_3PO_4 ($^{31}P\{^{1}H\}$). Elemental analyses were performed on a Perkin–Elmer 2400 CHN microanalyser.

2.4. *Crystal structure determination and refinement*

The X-ray data for **1** were collected at −173 °C using a KM4CCD diffractometer and graphite-monochromated Mo Kα radiation generated from Diffraction X-ray tube operated at 50 kV and 35 mA. The images were indexed, integrated and scaled using the KUMA data reduction package [10]. The structure was solved by direct method using SHELXS97 and refined by the full-matrix least-squares method on all F^2 data [11]. Non-hydrogen atoms were included in the refinement, with anisotropic displacement parameters. The hydrogen atoms were positioned geometrically and refined isotropically. The hydrogen atoms of NH groups were included from $\Delta\rho$ maps and fixed at a distance 0.92 and refined isotropically. Calculations were preformed using WINGX program [12].

*trans-[Pd(HL$^{I}_{2}$–S)$_2$Cl$_2$] (**1**).* $C_{22}H_{50}Cl_2N_4O_6P_2PdS_2$, M_r = 770.02 g mol^{-1}, yellow prisms, monoclinic, space group $P2_1/c$, a = 10.517(4), b = 17.316(5), c = 9.774(4) Å, β = 99.22(4)°, V = 1757.0(11) $Å^3$, Z = 2, D_c = 1.456 g cm^{-3}, μ(Mo Kα) = 0.929 mm^{-1}, reflections: 12196 collected, 4171 unique, R_{int} = 0.032, R_1(all) = 0.0285, wR_2(all) = 0.0605.

3. Results and discussion

3.1. *Synthesis*

N-Phosphorylated thioureas were prepared as previously described [9] and purified by recrystallization from dichloromethane–*n*-hexane mixtures.

Complexes **1–5** were prepared by the following procedure: $Pd(PhCN)_2Cl_2$ was dissolved in acetonitrile and added to a solution containing the corresponding ligand in the same solvent (Scheme 1). The compounds obtained are crystalline solids that are soluble in most polar solvents.

The molecular structures of complexes **1**–**5** were investigated by IR, ^{1}H, $^{31}P\{^{1}H\}$ NMR spectroscopy and elemental analysis. The crystal structure of **1** was established by single crystal X-ray diffraction analysis.

3.2. *IR and NMR spectroscopy*

The IR spectra of complexes **1**–**3** in Nujol are similar. The absorption bands of the P=O group of the neutral forms **HL**$^{I–III}$

are shifted by approximately 10–15 cm^{-1} to high frequencies relative to the band of the parent ligands. This confirms their nonparticipation in chelate complex formation. There are two signals for the NH proton in the IR spectra of complexes in the areas 3082–3094 and 3203–3217 cm^{-1} which are the characteristic range for amide protons participating in hydrogen bonds [4,7]. In the spectra of **1–3** there is an intense absorption band at 1515–1549 cm^{-1} corresponding to the S=C–N fragment. Besides very strong absorption bands for the POC group at 1008 cm^{-1}, there is a band of the COC group at 1140 cm^{-1} in the IR spectrum of **3**, containing crown-ether fragments. Presence of a wide absorption band of the POC fragment in the spectra of **1** and **2** in the area 998–1002 cm^{-1} also unequivocally confirms the substituted phosphoric fragment.

Application of the same procedure using the thioureas **HL**^{IV} and **HL**^{V} did not result in isolation of products with neutral forms of the corresponding ligand (Scheme 2). Complexes **4** and **5** obtained contain the central cation coordinated in a square-planar fashion by the C=S sulfur atoms and the P=O oxygen atoms of two anionic ligands. According to the IR, ^{1}H, ^{31}P{^{1}H} NMR spectroscopy, elemental analyses and melting point data complexes **4** and **5** are found to be the same as it was synthesized using the potassium salts of the thioureas **HL**^{IV} and **HL**^{V} in the aqueous ethanol medium [7].

The ^{31}P{^{1}H} NMR spectra of complexes **1–3** contain two singlet signals at −6.8 to −5.6 ppm, and a down-field shift, relative to that of the free ligands **HL**$^{I–III}$, is observed. This confirms the preservation of neutral forms of ligands [4]. Two different signals correspond to the phosphorus atoms with *cis*- and *trans*-configuration of ligands in the structure of complexes in the solution.

The ^{1}H NMR spectra of **1–3** contain a double set of signals. This also confirms the presence of *cis*- and *trans*-complexes in the solution. The signals for the methyl protons are observed at 1.38–1.53 ppm. The signals at 4.58–4.90 ppm correspond to the OCH protons. The aromatic proton signals in complex **2** are at 7.23–7.68 ppm and shifted down-field relative to that in complex **3**. In the ^{1}H NMR spectrum of **3** signals for the CH_2 protons, corresponding to the crown-ether fragment, are in the area 3.65–4.21 ppm.

There is a double set of signals for the NH protons in the ^{1}H NMR spectra of complexes **1–3** in the areas 9.61–10.16 and 10.37–10.83 ppm. The high-field shifted signals for the P(O)NH group are shown as a doublet with a characteristic constant $^{2}J_{PNH}$ = 5.5–5.8 Hz. The low-field signals correspond to the RNH protons. Both set of signals are low-field shifted relative to that in parent ligands [9] because of the intramolecular hydrogen bonds formation of the RNH···O=P and PNH···Cl–Pd types.

The unrecrystallized, pure complexes **1–5** are presumed to be single isomers, in view of the sharp melting points, respectively. However, from the ^{31}P{^{1}H} NMR spectra of fresh $CDCl_3$ solutions of unrecrystallized complexes **1–3**, it appears that *trans*-isomers predominate. The ^{31}P{^{1}H} NMR spectra of **1–3** remain invariant for 72 h, suggesting that the isomer distribution rapidly reaches equilibrium on dissolution of the compound. By contrast to complexes **1–3**, the ^{31}P{^{1}H} NMR spectra of **4** and **5** contain the unique singlet signals in the region characteristic to the deprotonated *N*-phosphorylated thioureas and thioamides [4]. The signals observed correspond to the *cis*-isomer [7]. Moreover, the ^{31}P{^{1}H} NMR spectra of **4** and **5** remain invariant for 72 h, suggesting that the *trans*-isomer is not observed in the solution of $CDCl_3$.

3.3. Crystal structure of 1

Crystals of **1** were obtained by slow evaporation of the solvent from dichloromethane–*n*-hexane solution of the complex.

The crystal structure of the *trans*-[Pd(HL^{I}-*S*)$_2$Cl$_2$] complex **1** is shown in Fig. 1, while the geometrical data such as bond distances, angles, torsion angles and hydrogen bonds are given in Tables 1 and 2.

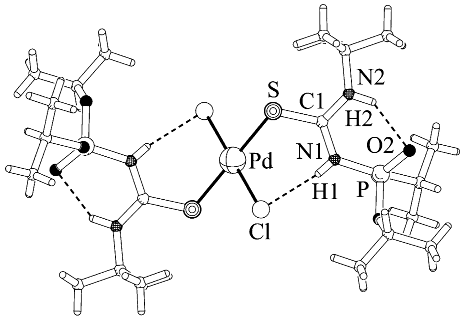


Fig. 1. Molecular structure of complex **1**.

Table 1
Selected bond lengths (Å), and bond angles (°) in complex **1**

Bond lengths			
Pd–Cl	2.3152(11)	P–N(1)	1.674(2)
Pd–S	2.3305(11)	N(1)–C(1)	1.370(3)
S–C(1)	1.708(2)	N(2)–C(1)	1.328(3)
P–O(2)	1.469(2)		
Bond angles			
Cl–Pd–S	85.31(4)	O(2)–P–N(1)	112.07(9)
Cl–Pd–Cl(A)	180.00	P–N(1)–C(1)	126.75(14)
Cl–Pd–S(A)	94.69(4)	S–C(1)–N(1)	120.82(15)
S–Pd–S(A)	180.00	S–C(1)–N(2)	121.71(15)
Pd–S–C(1)	113.86(8)	N(1)–C(1)–N(2)	117.5(2)
Torsion angles			
C(1)–S–Pd–Cl	128.09(9)	S–C(1)–N(1)–P	−170.94(11)
C(1)–S–Pd–Cl(A)	−51.91(9)	N(2)–C(1)–N(1)–P	10.5(3)
N(1)–C(1)–S–Pd	19.4(2)	C(1)–N(1)–P–O(2)	−38.1(2)
N(2)–C(1)–S–Pd	−162.02(14)		

Table 2
Parameters of hydrogen bonds for complex **1** [bond lengths (Å), and bond angles (°)]

N–H···A	*d*(N–H)	*d*(H···A)	D(N···A)	∠(NHA)
N(1)–H(1)···Cl	0.87(2)	2.26(2)	3.113(2)	167(2)
N(2)–H(2)···O(2)	0.90(2)	2.04(2)	2.832(3)	146(2)

Inspection of the structure shows the expected square-planar coordination with two ligands **HL**^{I} bonded through the sulfur atoms with Pd–S 2.316(1) Å. The bond lengths Pd–Cl are 2.442(1) Å, while the bond angles S(A)–Pd–Cl 95.54(3)° and S–Pd–Cl 85.46(3)° deviate significantly from 90°. The distortion being induced by the two hydrogen bonds between N1(H1)···Cl(A) and N1A(H1A)···Cl at 3.924 Å. Similar N(H)···Cl and N(H)···Br hydrogen bonds, and the resultant distortion of bond angles from 90°, have previously been observed for the *cis*-bis(*N*-benzoyl-*N*′-propylthioureato)dichloroplatinum(II) (**6**) and *trans*-bis(*N*-benzoyl-*N*′-propylthioureato)dibromopalladium(II) (**7**) complexes [3]. An additional hydrogen bond between the N2(H2) and the phosphoryl oxygen atom O2 of the coordinated ligand at 2.601(4) Å effectively locks the ring C1–N1–P–O2–H2–N2 into a planar structure. The structure, bond lengths and angles of the coordinated **HL**^{I} in *trans*-[Pd(HL^{I}-*S*)$_2$Cl$_2$] complex **1** are similar to that of the unbound RNHC(S)NHP(O)(O*i*Pr)$_2$ (R = alkyl, aryl) [4], as well as that of the coordinated ligand corresponding *cis*-platinum **6** and *trans*-palladium **7** complexes [3]. This mode of coordination of *N*-phosphorylated thiourea once again highlights the importance and relative stability of the intramolecular hydrogen bond between the thiourea NH group and the phosphoryl oxygen atom of this ligands in determining the preferred mode of S atom coordination.

4. Conclusions

The data presented allow to confirm that in the case of the investigated *N*-phosphorylthiourea ligands $\mathbf{HL}^{\mathrm{I-III}}$, containing secondary nitrogen at the *N*-thioacylamidophosphate moiety $C(S)NHP(O)(O\textit{i}Pr)_2$ (Scheme 1), the pure *trans*-$Pd(HL^{I-III}-S)_2Cl_2$ complexes were obtained by reaction with palladium(II). These complexes undergoes partial isomerization in solution to yield *cis*/*trans* distribution. In contrast the palladium(II) complexes with thioureas $\mathbf{HL}^{\mathrm{IV,V}}$, containing tertiary nitrogen at the *N*-thioacylamidophosphate moiety, under the same synthesis conditions forms *cis*-$Pd(L^{IV,V}-O,S)_2$ chelate complexes with deprotonated forms of the ligands both in solution and solid state.

Thus, the formation of the intramolecular hydrogen bonds $P{=}O\cdots HNR$ is a necessary condition for the monodentate *S*-isomer stabilization in a square-planar complexes of Pd(II). When H-bonding in the coordinated ligands is not possible the deprotonation of them and a change of the coordination mode in the Pd(II) complexes occurs.

5. Supplementary data

CCDC 669887 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic 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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