Synthesis and Characterization of Palladium and Platinum Complexes of N,N'-Bis[2'-(diphenylphosphino)phenyl]propane-1,3-diamine. Single-Crystal Structures of [Pd(Ph₂PC₆H₄NC₃H₆NC₆H₄PPh₂)] and [Pt(Ph₂PC₆H₄NH)(SEt₂)Cl]

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Received January 26, 1998

Novel mononuclear metal complexes with the formula [M(Ph₂PC₆H₄NC₃H₆NC₆H₄PPh₂)] (M=Pd (**1**); M=Pt (**2**)) were obtained when N,N'-bis[2'-(diphenylphosphino)phenyl]propane-1,3-diamine, **I** was mixed with *cis*-dichlorobis(diethylsulfide)palladium and platinum in the presence of NEt₃. Two mononuclear metal compounds with the formula [M(Ph₂PC₆H₄NH)(SEt₂)Cl] (M=Pd (**3**); M=Pt (**4**)) were synthesized from M(SEt₂)₂Cl₂ and N-(2'-diphenylphosphinophenyl)-4-amino-1,1,1,5,5,5-hexafluoro-3-penten-2-one, **II** by the elimination reaction of hexafluoro pentenone. The X-ray single crystal structures of **1** and **4** are described. X-ray single crystal diffraction analyses reveal that compound **1** is a mononuclear palladium compound with P,N,N,P-coordination mode and **4** is a mononuclear platinum compound with P,N-coordination mode.

Introduction

Neutral polydentate ligands can exhibit a wild variety of coordination behaviors, which can influence stereochemistry and other properties of the resulting complexes. A characteristic of the coordination chemistry of chelating bis(tertiaryphosphines) is their distinctive ability to stabilize metal ions in a variety of uncommon oxidation states and geometries.[1] On the other hand, diamine ligands are also known to stabilize metal ions in a variety of low and high oxidation states, and their metal complexes are powerful catalysts in both biological and industrial roles.[2] Accordingly, bidentate ligands containing both nitrogen and phosphorous donor atoms are of special importance since they utilize both σ-donating and π-accepting atoms in the same ligand and retain many of the properties of amines and phosphines.[3] (o-Diphenylphosphinophenyl)amine, **I** is the first example of a hybrid ligand containing both a hard amine and a soft phosphine.[4] We have been interested in the coordination chemistry of the bidentate (P,S⁵- and Si, Si⁶-type), terdentate (N,N,S⁷- and P,N,S⁸-type), and tetradentate ligands(N,S,N,S⁹- and P,N,N,P¹⁰-type). In this paper we wish to extend our studies to terdentate (P,N,O type) (**II**) and tetradentate ligand (P,N,N,P type¹¹) (**III**) containing the (2-aminophenyl)phosphino subunit. We now describe the interaction of the *cis*-dichlorobis(diethylsulfide) palladium and platinum with P,N,O- and P,N,N,P-type ligands.

I   II   III

Experimental Section

General Procedures. All manipulations were performed under a dry and oxygen-free dinitrogen or argon atmosphere using standard Schlenk techniques or in a HE-493 dry box. Toluene, hexane, and THF were freshly distilled from sodium/benzophenone prior to use. All ¹H and ³¹P NMR spectra were recorded using a Bruker AM-360 spectrometer. 1H NMR spectra were referenced against the residual ¹H impurity of the deuterated solvent and ³¹P NMR were referred to 85% H₃PO₄ as an external standard. IR spectra were recorded on a Shimadzu FT IR-8501 spectrometer. Mass spectra were recorded on a high resolution VG 70-VSEG instrument, and elemental analyses were performed by the Basic Science Center. The ligands [Ph₂PC₆H₄-NHC₃H₆NHC₆H₄PPh₂], [Ph₂PC₆H₄NHC(CF₃)CHC(O)CF₃],¹² and the compounds [M(SEt₂)₂Cl₂] (M=Pd, Pt) were prepared according to the literature methods.¹³

Preparation of [Pd(Ph₂PC₆H₄NC₃H₆NC₆H₄PPh₂)] (1). A toluene solution (25 mL) of Pd(SEt₂)₂Cl₂ (0.26 g, 0.74 mmol) was added to a toluene solution (25 mL) of Ph₂PC₆H₄NHC₃H₆NHC₆H₄PPh₂ (0.48 g, 0.80 mmol) dropwise at room temperature. NEt₃ (0.1 mL) was added to the reaction mixture. The solution was stirred at room temperature for 1 h and refluxed for 5 h. The volatiles were removed under vacuum to afford an orange solid, which was extracted with toluene (20 mL) and cooled to −25 °C for 24 h. The orange solid was collected by filtration, washed with pentane, and dried under vacuum (0.42 mg, 82%). Crystals suitable for X-ray crystallography were obtained by recrystallization from toluene/pentane. mp 228-232 °C. ¹H NMR (C₆D₆): δ 7.6-6.2 (18H, m, aromatic), 2.82 (4H, br. t, N-CH₂), 1.84 (2H, m, -CH₂-). ³¹P NMR (C₆D₆): δ 42.32. UV (CH₂Cl₂, λₘₐₓ, ε) 324 (1.05×10⁴), 423 (3.3×10³), 480 (1.8×10³). MS (EI): m/z 698 [M⁺]. Anal. Calcd. for C₃₉H₃₄N₂P₂Pd: C, 67.05; H, 4.87. Found: C, 67.72; H, 5.02.

Preparation of [Pt(Ph₂PC₆H₄NC₃H₆NC₆H₄PPh₂)]
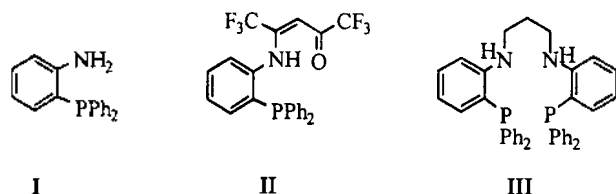
(2). 2 was prepared according to the same method as in **1** except that Pt(SEt$_2$)Cl$_2$ was used as a starting material. Yield: 86%. mp 258 °C (dec.). ^{1}H NMR (C$_6$D$_6$): δ 8.0-6.0 (18H, m, aromatic), 2.62 (4H, br. t, N-CH$_2$), 1.76 (2H, m, -CH$_2$-). ^{31}P NMR (C$_6$D$_6$): δ 25.74 (J_{Pt-P}=3184 Hz). MS (EI): m/z 787 [M$^+$]. Anal. Calcd. for C$_{39}$H$_{34}$N$_2$P$_2$Pt: C, 59.47; H, 4.32. Found: C, 58.74; H, 4.24.

Preparation of [Pd(Ph$_2$PC$_6$H$_4$NH)(SEt$_2$)Cl] (3). A toluene solution (20 mL) of Pd(SEt$_2$)$_2$Cl$_2$ (0.21 g, 0.59 mmol) was added to a toluene solution (20 mL) of Ph$_2$PC$_6$-H$_4$NHC(CF$_3$)CHC(O)CF$_3$ (0.30 g, 0.64 mmol) at room temperature. NEt$_3$ (0.1 mL) was added to the solution. The solution was stirred at room temperature for 1 h and refluxed for 4 h. The volatiles were removed under vacuum to afford a red solution, which was extracted with toluene (20 mL) and cooled to −25 °C overnight. The orange-red solid was collected by filtration and dried under vacuum. This product was recrystallized from toluene/pentane at −10 °C. Yield: 72%. mp 194-198 °C. ^{1}H NMR (C$_6$D$_6$): δ 8.32 (1H, s, NH), 7.8-6.3 (14H, m, aromatic), 1.44 (4H, q, J_{HH}=7.42 Hz, S-CH$_2$), 0.84 (6H, t, J_{HH}=7.42 Hz, CH$_3$). ^{31}P NMR (C$_6$D$_6$): δ 53.92. IR (on KBr pellet; cm^{-1}): v(N-H) 3428. MS (EI): m/z 508 [M$^+$]. Anal. Calcd. for C$_{22}$H$_{25}$NPClPdS: C, 51.97; H, 4.92. Found: C, 52.44; H, 5.22.

Preparation of [Pt(Ph$_2$PC$_6$H$_4$NH)(SEt$_2$)Cl] (4). 4 was prepared as **3** except that Pt(SEt$_2$)$_2$Cl$_2$ was used as a starting material. Yield: 72%. mp 206-210 °C. ^{1}H NMR (C$_6$D$_6$): δ 8.18 (1H, s, NH), 8.0-6.2 (14H, m, aromatic), 1.40 (4H, q, J_{HH}=7.28 Hz, S-CH$_2$), 0.82 (6H, t, J_{HH}=7.28 Hz, CH$_3$). ^{31}P NMR (C$_6$D$_6$): δ 37.48 (J_{Pt-P}=3014 Hz). IR (on KBr pellet; cm^{-1}): v (N-H) 3384. UV (CH$_2$Cl$_2$, λ_{max}, ε) 344 (2.84×10^3), 452 (6.8×10^2). MS (EI): m/z 597 [M$^+$]. Anal. Calcd. for C$_{22}$H$_{25}$NPClPtS: C, 44.22; H, 4.19. Found: C, 45.06; H, 3.88.

X-ray Crystallography. Details of the crystal data

Table 1. Crystallographic data for the structural studies of compound **1** and **4**

empirical formula	**1**	**4**
	C$_{39}$H$_{34}$N$_2$P$_2$Pd	C$_{22}$H$_{25}$NPClPtS
fw	701.07	597.02
cryst class	triclinic	monoclinic
space group	P1̄	P2$_1$/n
a, Å	12.15(3)	10.658(9)
b, Å	17.58(2)	16.049(1)
c, Å	9.87(2)	13.576(2)
α, deg	99.9(2)	
β, deg	100.6(2)	108.27(3)
γ, deg	75.6(1)	
V, Å^3	1990(7)	2205(2)
D_c, gcm^{-3}	1.170	1.798
$F(000)$	720	1160
Z	2	4
μ (Mo Kα), cm^{-1}	5.63	67.21
no. of reflns collctd	7345	4254
no. of reflns obsd	6984	4028
goodness of fit	1.86	1.36
R1^a	0.066	0.034
wR2^a	0.074	0.038

^{a} R1=Σ ‖ F_o|-|F_c ‖ /Σ|F_o|; wR2={Σ[w(F_0^2-F_c^2)^2]/Σ[wF_0^2]}$^{1/2}$.

and a summary of intensity data collection parameters for **1** and **4** are given in Table 1. The crystals of **1** and **4** were grown from a toluene/pentane solution at −15 °C. The crystals of **1** and **4** were mounted in thin-walled glass capillaries and sealed under argon. The data sets of **1** and **4** were collected using a Rigaku AFC6S employing graphite-monochromated MoKα radiation (λ=0.71069 Å) at −120 °C. The structures were solved by direct methods. Refinements were made by full-matrix least-squares techniques based on F to minimize the quantity Σw(|F_o|-|F_c|)^2 with w=1/σ^2(F). Non-hydrogen atoms of **1** and **4** were anisotropically refined, and hydrogen atoms were isotropically refined. The hydrogen atoms of **1** and **4** were included as constant contributions to the structure factors. The refinement of **1** converged to R1=0.066 and wR2=0.074 and that of **4** converged to R1=0.034 and wR2=0.038. Selected bond distances and angles for **1** and **4** are given in Table 2 and 3, respectively. The molecular structure of compounds **1** and **4** are given in Figures 1 and 2, respectively.

Results and Discussion

Synthesis and Characterization of [M(Ph$_2$PC$_6$H$_4$-NC$_3$H$_6$NC$_6$H$_4$PPh$_2$)] (M=Pd, Pt). The reaction of N,N'-bis[2'-(diphenylphosphino)phenyl]propane-1,3-diamine with

Table 2. Selected bond lengths (Å) and angles (deg) for compound **1**

Pd(1)-P(1)	2.251(6)	P(2)-C(10)	1.78(1)
Pd(1)-P(2)	2.244(7)	P(2)-C(28)	1.83(1)
Pd(1)-N(1)	2.06(1)	P(2)-C(34)	1.84(1)
Pd(1)-N(2)	2.05(1)	N(1)-C(1)	1.45(2)
P(1)-C(4)	1.79(1)	N(1)-C(9)	1.37(2)
P(1)-C(16)	1.81(1)	N(2)-C(3)	1.48(2)
P(1)-C(22)	1.82(1)	N(2)-C(15)	1.36(2)
P(1)-Pd(1)-P(2)	100.1(2)	C(4)-P(1)-C(22)	106.0(6)
P(1)-Pd(1)-N(1)	83.0(4)	C(16)-P(1)-C(22)	107.8(6)
P(1)-Pd(1)-N(2)	177.6(3)	Pd(1)-P(2)-C(10)	102.2(5)
P(2)-Pd(1)-N(1)	176.9(3)	Pd(1)-P(2)-C(28)	119.8(5)
P(2)-Pd(1)-N(2)	82.3(4)	Pd(1)-P(2)-C(34)	114.6(5)
N(1)-Pd(1)-N(2)	94.7(5)	C(10)-P(2)-C(28)	107.2(6)
Pd(1)-P(1)-C(4)	101.2(5)	C(10)-P(2)-C(34)	105.2(6)
Pd(1)-P(1)-C(16)	114.9(5)	C(28)-P(2)-C(34)	106.7(6)
Pd(1)-P(1)-C(22)	119.4(4)	Pd(1)-N(1)-C(1)	123.6(9)
C(4)-P(1)-C(16)	106.1(6)	Pd(1)-N(1)-C(9)	119.8(8)

Table 3. Selected bond lengths (Å) and angles (deg) for compound **4**

Pt(1)-Cl(1)	2.352(2)	S(1)-C(21)	1.820(8)
Pt(1)-S(1)	2.299(2)	P(1)-C(1)	1.809(8)
Pt(1)-P(1)	2.212(2)	P(1)-C(7)	1.817(8)
Pt(1)-N(1)	2.000(7)	P(1)-C(13)	1.807(8)
S(1)-C(19)	1.841(9)	N(1)-C(6)	1.36(1)
Cl(1)-Pt(1)-S(1)	93.6(1)	Pt(1)-S(1)-C(19)	109.4(3)
Cl(1)-Pt(1)-P(1)	171.04(8)	Pt(1)-S(1)-C(21)	107.1(3)
Cl(1)-Pt(1)-N(1)	88.7(2)	Pt(1)-P(1)-C(1)	102.5(3)
S(1)-Pt(1)-P(1)	94.54(9)	Pt(1)-P(1)-C(7)	114.6(3)
S(1)-Pt(1)-N(1)	177.2(2)	Pt(1)-P(1)-C(13)	119.1(3)
P(1)-Pt(1)-N(1)	83.3(2)	Pt(1)-N(1)-C(6)	122.5(6)
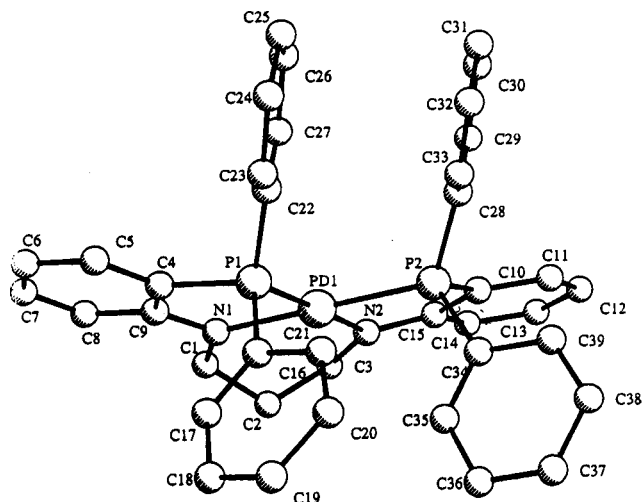
Figure 1. Molecular structure of [Pd(Ph₂PC₆H₄NC₃H₆NC₆H₄PPh₂)] (1).
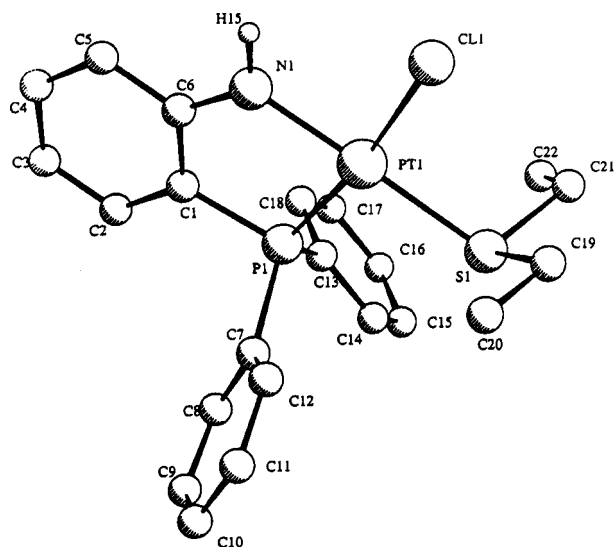
Figure 2. Molecular structure of [Pt(Ph₂PC₆H₄NH)(SEt₂)Cl] (2).

cis-dichlorobis (diethylsulfide)palladium or platinum in the presence of NEt₃ affords the corresponding metal complexes, in which two hydrogen atoms have been lost from the *aza* groups *via* the hydrogen chloride elimination reaction (eq. 1).
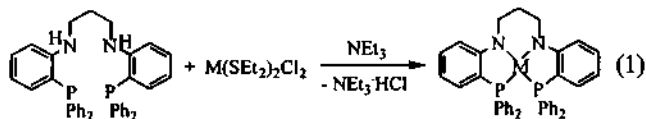
The resulting orange compounds 1 and 2 were isolated as air-stable crystalline solids in a high yield. Theses compounds are soluble in common organic solvents. The complexes 1 and 2 have been characterized by ¹H and ³¹P NMR, MS, and elemental analyses. The structure of compound 1 was determined based on its X-ray crystal structure. A singlet resonance in the ³¹P{¹H} NMR spectrum of 1 establishes the equivalent phosphines and suggests a *cis* geometry. For the platinum complex 2 the expected pattern

due to its A₂X spin system was observed with coupling constant ¹*J*(Pt-P)(3184 Hz) similar to those for [PtL]²⁺ (L=1,3-bis(*o*-amino phenyl)phenylphosphino)propane)¹⁴ and [PtCl (Buᵗ₂PCH₂CH₂C=CHCH₂PBuᵗ₂)].¹⁵ The large value in JPt-P is typical for a phosphorous atom *trans* to an amino nitrogen donor. The mass spectra of the mononuclear complexes 1 and 2 show molecular ion clusters at m/e 698 and 787 for Pd and Pt, respectively. While all the spectroscopic data and elemental analyses were in good agreement with the proposed structure, confirmation of the mononuclear structure of 1 was provided by X-ray crystallography.

Synthesis and characterization of [M(Ph₂PC₆H ₄NH)(SEt₂)Cl] (M=Pd, Pt). Initially, we expected that the reactions of N-(2'-diphenylphosphinophenyl)-4-amino-1, 1,1,5,5,5-hexafluoro-3-penten-2-one with M(SEt₂)Cl₂ (M=Pd, Pt) would give the mononuclear complexes with a P, N, O terdentate mode. Unexpectedly, the reaction of the ligand II with M(SEt₂)Cl₂ (M=Pd, Pt) led to the cleavage of N-C bond, affording the complexes as in eq. 2.
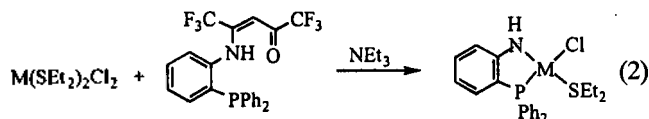
The resulting orange compounds 3 and 4 were isolated as air-stable crystalline solids in a high yield and soluble in benzene, toluene, and THF. The initial indication of a mononuclear formulation for 3 and 4 stemmed from the observation of a parent ion in the mass spectrum at m/z 508 and 597, respectively. The peak at 3428 cm⁻¹ in 3 is assigned to the stretching mode of NH. The ¹H NMR spectrum of 3 shows one peak at 8.32 ppm due to the hydrogen atom of the N-H group. The ³¹P NMR spectrum of 3 shows one peak at 53.92 ppm. Although all the spectroscopic data demonstrate the absence of hexafluoropentenone moiety from the ligand II, the mononuclear structure of 4 with exclusion of hexafluoropentenone was confirmed by X-ray crystallography.

Description of the Molecular Structure of [Pd(Ph₂-PC₆H₄NC₃H₆NC₆H₄PPh₂)]. Crystals of 1 suitable for an X-ray diffraction study were grown from toluene/pentane at −15 °C, and the structure of 1 was determined from data collected at −120 °C. A summary of data collection and crystallographic parameters are given in Table 1. Selected bond lengths and angles are given in Table 2. An ORTEP diagram of the solid state structure giving the atom-numbering scheme is given in Figure 1. The molecule contains both two PdNC₂P five-membered rings and one PdN₂C₃ six-membered ring. The palladium atom Pd(1) is found in the usual four-coordinate square-planar coordination geometry. The ligand I was found to behave as a P₂N₂ tetradentate ligand such that it has an approximate C₂ axis along the bisector of the P-Pd-P angle. The chelate bite angles are 83.0(4)° for P(1)-Pd(1)-N(1) and 82.3(4)° for P(2)-Pd(1)-N (2), which are not unusual for chelate rings of this type, but they are smaller than ideal square-planar angles and result in large P(1)-Pd(1)-P(2) angle of 100.1(2)°. This angular disturbance from the ideal square-planar geometry emphasize the constraints of the chelate ring. These angles, however, imply that Pd atom is exactly on the plane of P₂N₂.

The Pd-P distances range from 2.244(7) to 2.251(6) Å

and the average vale of 2.247 Å is somewhat shorter than the range of value 2.28-2.31 Å, typically observed for square-planar Pd(II) complexes having two bidentate phosphorous ligands.[16] Pd(II)-P distances are rather insensitive to the geometry at the metal, but it has been suggested that they are sensitive to the steric crowding.[17] The Pd-N distances, 2.06(1) and 2.05(1) Å are not significantly different from each other. The Pd-N (amido) distance is shorter than the Pd-N (amine) distance (2.20-2.35 Å), a feature consistent with the literature.

Description of the molecular Structure of [Pt(Ph₂-PC₆H₄NH)(SEt₂)Cl]. A crystal of **4** suitable for X-ray diffraction study was grown from toluene/pentane at −15 °C. A summary of the data collection and crystallographic parameters for **4** are given in Table 1. The relevant bond lengths and angles are shown in Table 3. The molecular structure of **4** is given in Figure 2. The molecule contains one PtNC₂P five-membered ring. In the solid state, the Pt(1) atom is coordinated by one nitrogen, one phosphorus, one sulfur, and one chloride atom in a distorted square-planar configuration. As the N(1)-Pt(1)-S(1) and P(1)-Pt(1)-Cl(1) bond angle are 177.2(2)° and 171.04(8)°, respectively, the coordination environment about Pt(1) may be described as a distorted square-planar. The Pt(1)-N(1) bond distance (2.000(7) Å) is slightly shorter than that of Pt-N bond (2.063(7) Å) in Pt(o-Ph₂PC₆H₄NC(O)Ph)₂[18] which falls within the range of 2.02(1)-2.09(2) Å found for the small number of such complexes which have been structually characterized.[19] The relative short bond distance in Pt(1)-S (1) (2.299(2) Å) compared with that of Pt-S bond (2.439(2) Å) in Pt(MeL)Cl₃[20] may be attributable to the back bonding.

The use of the N,N'-bis[2'-(diphenylphosphino)phenyl] propane-1,3-diamine and N-(2'-diphen ylphosphinophenyl)-4-amino-1,1,1,5,5,5-hexafluoro-3-penten-2-one as ligands in the organometallic chemistry of palladium and platinum leads to mononuclear complexes of these metals having a tetradentate P₂N² mode or a bidentate PN mode. X-ray crystallographic studies and spectroscopic data confirm that these complexes represent square-planar geometry. Our continuing investigations are oriented concerning the studies of unusual ligands and their organometallic complexes.

Supporting Information Available. Tables of bond distances and angles, atomic coordination, fractional coordinates, thermal parameters, and least-square results for **1** and **4** (34 pages) are available from one of the author (Ko, J.) upon request.

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