Synthesis, Reactivity, and Crystal Structure of the η^2-Thiocarbamoyl Palladium Complex $[Pd(PPh_3)_2(\eta^2\text{-}SCNMe_2)][PF_6]$

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The η^1-thiocarbamoyl palladium complexes $[Pd(PPh_3)(\eta^1\text{-}SCNMe_2)(\eta^2\text{-}S_2R)]$ (R = $P(OEt)_2$, **2**; $CNEt_2$, **3**) and *trans*-$[Pd(PPh_3)_2(\eta^1\text{-}SCNMe_2)(\eta^1\text{-}Spy)]$, **4**, (pyS: pyridine-2-thionate) are prepared by reacting the η^2-thiocarbamoyl palladium complex $[Pd(PPh_3)_2(\eta^2\text{-}SCNMe_2)][PF_6]$, **1** with $(EtO)_2PS_2NH_4$, Et_2NCS_2Na, and pySK in methanol at room temperature, respectively. Treatment of **1** with dppm (dppm: bis(diphenylphosphino)methane) in dichloromethane at room temperature gives complex $[Pd(PPh_3)(\eta^1\text{-}SCNMe_2)(\eta^2\text{-}dppm)][PF_6]$, **5**. All of the complexes are identified by spectroscopic methods and complex **1** is determined by single-crystal X-ray diffraction.

Keywords: Thiocarbamoyl; Palladium, Crystal structure; Dithiocarbamate; Phosphorodithioate ligand.

INTRODUCTION

The study of transition-metal compounds containing the N,N-dialkylthiocarbamoyl ligand ($^-SCNR_2$) is of interest in that the ligand may act as a mono- or bidentate ligand, resulting in novel structural and chemical features.[1] The investigation of transition-metal complexes with thiocarbonyl containing ligands[2] and palladium derivatives are of interest because these systems may find applications as new homogeneous catalysts. Recently we have shown[3] the dissociation of either the chloride or the triphenylphosphine ligand of complex $[Pd(PPh_3)_2(\eta^1\text{-}SCNMe_2)(Cl)]$ to form complex $[Pd(PPh_3)_2(\eta^2\text{-}SCNMe_2)][Cl]$ or the dipalladium complex $[Pd(PPh_3)Cl]_2(\mu,\eta^2\text{-}SCNMe_2)_2$.

To understand the electrophilic reactivity and the coordination mode of the Me_2NCS ligand, herein, we report the results of thio and diphos ligands addition to complex $[Pd(PPh_3)_2(\eta^2\text{-}SCNMe_2)][PF_6]$ and its crystal structure.

RESULTS AND DISCUSSION

In an early study,[3] we described the synthesis and X-ray crystallography of the η^1-thiocarbamoyl palladium complex $[Pd(PPh_3)_2(\eta^1\text{-}SCNMe_2)(Cl)]$. Treatment of $[Pd(PPh_3)_2(\eta^1\text{-}SCNMe_2)(Cl)]$ with NH_4PF_6 in acetone at room temperature resulted in chloride abstraction reaction, affording the η^2-thiocarbamoyl palladium complex $[Pd(PPh_3)_2(\eta^2\text{-}SCNMe_2)][PF_6]$, **1** with 92% isolate yield (Scheme I). The air-stable yellow compound **1** is soluble in DMSO, CH_2Cl_2 and CH_3CN and insoluble in MeOH, *n*-hexane, and diethyl ether. The analytical data of **1** are in good agreement with the formulation. In the FAB mass spectra, one base peak with the typical Pd isotope distribution is in agreement with the $[M^+ - PF_6]$ molecular mass of **1**. The IR spectrum of **1** shows four stretchings at 1630 cm^{-1} for C=N, at 1481 and 1437 cm^{-1} for C=S, and at 839 cm^{-1} for PF_6 group. In the ^1H NMR spectrum of **1**, the two methyl protons of the $SCNMe_2$ ligand exhibit two resonances at δ 2.45 and δ 3.61. In the $^{13}C\{^1H\}$ NMR spectrum of **1**, two singlet resonances appear at δ 45.9 and δ 53.9 and one doublet resonance appears at δ 212.1 ($^2J_{P\text{-}C}$ = 6.7) for the carbon atom of the two methyl and thiocarbamoyl group, respectively. The $^{31}P\{^1H\}$ NMR spectrum of **1** shows two doublet resonances at δ 23.1 and δ 35.1 due to the chemical inequivalence of the two PPh_3 ligands and the relative downfield resonance compared to that of the starting complex $[Pd(PPh_3)_2(\eta^1\text{-}SCNMe_2)(Cl)]$ (δ 16.6) which shows the cationic character of **1**. From the description, it is clear that the palladium of **1** is side-on bound through the C-S moiety of the $SCNMe_2$ ligand.

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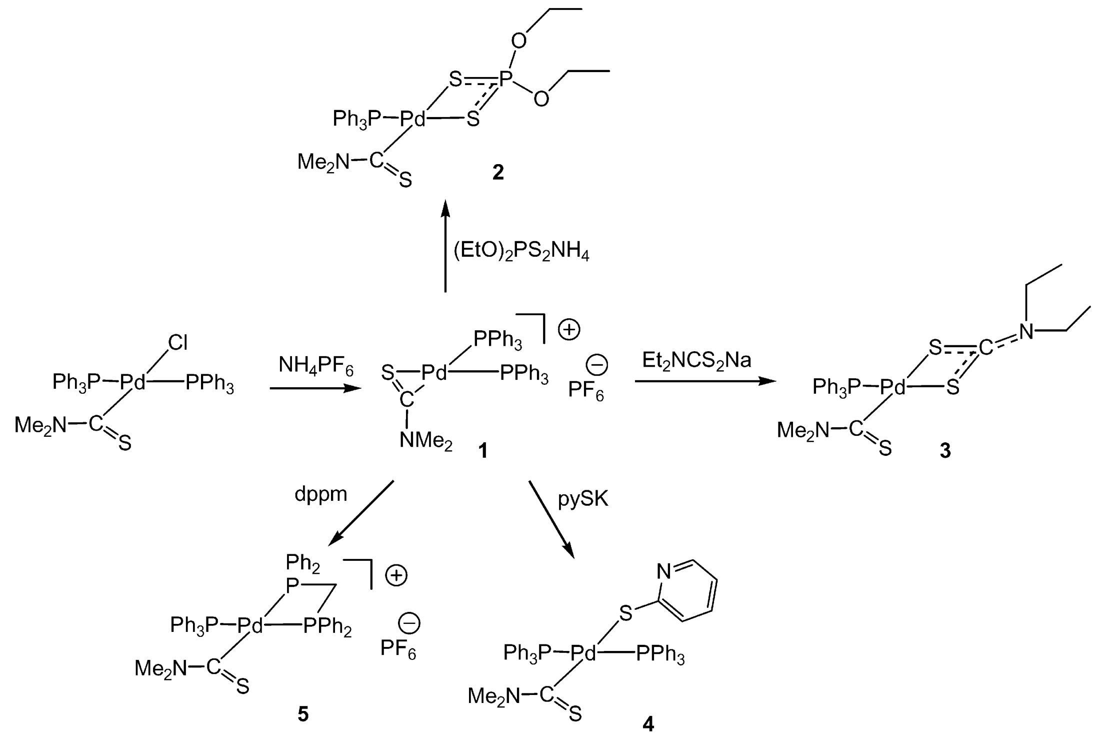
Scheme I



To confirm the η^2-thiocarbamoyl coordination mode, a single crystal of **1** suitable for X-ray diffraction study was grown by slow *n*-hexane diffusion into a dichloromethane solution at 4 °C. An ORTEP plot of **1** is shown in Fig. 1. In complex **1**, the Pd atom and its neighboring atoms, P(1), P(2), S(1) and C(1) lie in a distorted squared plane. The distortion is mainly due to the short bite of the C=S linkage [C-Pd-S, 44.54(16)°]. A least-squares plane calculation reveals the planarity of the P(2)P(1)C(1)S(1) core (largest deviation 0.031(1) Å). The C(1)-S(1) bond distance of 1.667(6) Å is similar to the corresponding carbon-sulfur bond distance observed in η^2-CS_2 (sp^2) transition-metal complexes [1.65(3) Å in [$(PPh_3)_2Pd(\eta^2$-$CS_2)$]].[4] The Pd-S(1) distance of 2.3243(5) Å is within the normal Pd-S length range (2.23~2.32 Å).[5] Within the $SCNMe_2$ ligand itself, the geometry is consistent with significant partial double bond character in the C-S and SC-N bond. Thus, the C-S bond distance (1.667(6) Å) is comparable to the C-S double bond in ethylenethiourea, although they are longer than those in free CS_2 (1.554 Å). The SC-N bond distance (1.303(7) Å) is typical for a C-N bond having partial double bond character and is certainly much shorter than the normal C-N (1.47 Å) single bond. The Me-N distance is normal for a single bond.

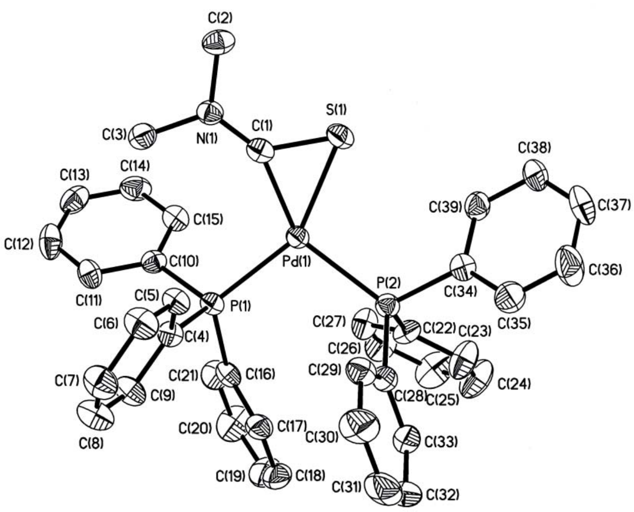


Fig. 1. An ORTEP drawing with 30% thermal ellipsoids and atom-numbering scheme for the cationic complex [$Pd(PPh_3)_2(\eta^2$-$SCNMe_2)$][PF_6], **1**.

Treatment of [$Pd(PPh_3)_2(\eta^2$-$SCNMe_2)$][PF_6], **1** with $(EtO)_2PS_2NH_4$ in methanol for 10 min yields complex [$Pd(PPh_3)(\eta^1$-$SCNMe_2)\{\eta^2$-$S_2P(OEt)\}$], **2** in 75% yield exclusively (Scheme I). Compound **2** is an air-stable, yellow solid and is readily soluble in polar organic solvents such as dichloromethane and acetonitrile but is insoluble in diethyl ether and *n*-hexane.

The identification of **2** as [$Pd(PPh_3)(\eta^1$-$SCNMe_2)(\eta^2$-

$S_2P(OEt)_2)]$ is according to the spectroscopic data. The FAB mass spectrum of **2** shows a base peak at *m/z* = 642 which corresponds to the molecular mass of **2**. In the ^1H NMR spectrum, the methyl protons and ethoxy protons are observed at δ 2.93, 3.30 and at δ 1.37, 4.24 (J_{H-H} = 6.95), respectively, and the corresponding resonances in the $^{13}C\{^1H\}$ NMR spectrum appear at δ 41.8, 47.1 and at δ 15.7, 64.2, 64.5, respectively. Notably, the relative down field $^{13}C\{^1H\}$ resonance of the carbon atom of the thiocarbonyl reveals the η^1-thiocarbamoyl property. The $^{31}P\{^1H\}$ NMR resonances of **2** appear at δ 100.4 (PS_2) and δ 23.9 (PPh_3) with a ratio of 1:1 from the integration of the $^{31}P\{^1H\}$ NMR spectra. From the above description, it is clear that the phosphorodithioate ligand, $(EtO)_2PS_2^-$, coordinated to the palladium through the two sulfur atoms.

The same procedure was used to prepare other thiocarbamoyl thio-complexes $[Pd(PPh_3)(\eta^1\text{-}SCNMe_2)(\eta^2\text{-}S_2CNEt_2)]$, **3** and *trans*-$[Pd(PPh_3)_2(\eta^1\text{-}SCNMe_2)(\eta^1\text{-}Spy)]$, **4** with 69% and 86% yields, respectively. In the FAB mass spectra, two base peaks with the typical Pd isotope distribution are respectively in agreement with the $[M^+ - BF_4]$ and the $[M^+ - pyS]$ molecular masses of **3** and **4**. The $^{31}P\{^1H\}$ NMR spectra of **3** (δ 25.3) and **4** (δ 20.9) are similar to that of the neutral complex **2**. The η^1-pyS mode of **4** was formed due to its chelating ability being less than those of the S_2CNEt_2 and $S_2P(OEt)_2$ ligands.

Treatment of $[Pd(PPh_3)_2(\eta^2\text{-}SCNMe_2)][PF_6]$, **1** with dppm in dichloromethane for 10 min yields complex $[Pd(PPh_3)(\eta^1\text{-}SCNMe_2)(\eta^2\text{-}dppm)][PF_6]$, **5** with 78% isolate yield (Scheme I). The FAB mass spectrum of **5** shows a base peak at *m/z* = 578 which corresponds to a fragment formed by loss of the PPh_3 group from **5**. The IR spectrum of **5** is similar to that of **1**. The ^1H NMR spectrum of **5** exhibits two singlet resonances at δ 2.15, 3.74 and one triplet resonance at δ 2.68 assignable to the N-methyl and P-methylene protons, respectively. The corresponding $^{13}C\{^1H\}$ NMR signals appear at δ 34.3, 45.2 and δ 30.9, respectively. In the $^{31}P\{^1H\}$ NMR spectrum of **5**, three doublets of doublet resonances at δ 4.51, 11.4 and δ 29.6 with ratios of 1:1:1 are assigned to the dppm and PPh_3 ligands, respectively, that indicates the η^2-P-coordination of the dppm ligand. Attempted reactions of complex **1** with KSCN and bipy gave no reaction but with $NH_2CH_2C\equiv CH$ gave more than ten complexes, although this reaction was not pursued further.

We employ thio and dppm ligands addition to the thiocarbamoyl palladium complex to investigate the thiocarbamoyl bonding modes. η^1-Thiocarbamoyl bonding mode is observed in the dithiophosphate, dithiocarbamate, and dppm ligands. Crystal structure of **1** confirms the η^2-thiocarbamoyl bonding mode.

EXPERIMENTAL SECTION

General Procedures

All manipulations were performed under nitrogen using vacuum-line and standard Schlenk techniques. NMR spectra were recorded on an AM-500 WB FT-NMR spectrometer and are reported in units of parts per million with residual protons in the solvent as an internal standard ($CDCl_3$, δ 7.24). IR spectra were measured on a Nicolate Avator-320 instrument and referenced to polystyrene standard, using cells equipped with calcium fluoride windows. MS spectra were recorded on a JEOL SX-102A spectrometer. Solvents were dried and deoxygenated by refluxing over the appropriate reagents before use. *n*-Hexane and diethyl ether were distilled from sodium-benzophenone. Acetonitrile and dichloromethane were distilled from calcium hydride, and methanol was distilled from magnesium. All other solvents and reagents were of reagent grade and used as received. Elemental analyses and X-ray diffraction studies were carried out at the Regional Center of Analytical Instruments located at National Taiwan University. $Pd(PPh_3)_4$ was purchased from Strem Chemical; $(EtO)_2PS_2NH_4$, Et_2NCS_2Na, pySH, and dppm were purchased from Merck.

Preparation of $[Pd(PPh_3)_2(\eta^2\text{-}SCNMe_2)][PF_6]$, 1

Acetone (20 mL) was added to a flask (100 mL) containing NH_4PF_6 (0.163 g, 1.0 mmol) and $[Pd(PPh_3)_2(\eta^1\text{-}SCNMe_2)(Cl)]$ (0.754 g, 1.0 mmol). The solution was stirred for 5 min at room temperature. The solvent was then removed under vacuum. The remaining solid was dissolved in 10 mL of CH_2Cl_2 and the solution was filtered to remove excess NH_4Cl. The solution was concentrated under vacuum and *n*-hexane (10 mL) was added to initiate precipitation. The yellow-orange solids **1** were formed which were isolated by filtration (G4), washed with *n*-hexane (2 × 10 mL) and subsequently dried under vacuum yielding $[Pd(PPh_3)_2(\eta^2\text{-}SCNMe_2)][PF_6]$, **1** (0.79 g, 92%). Further purification was accomplished by recrystallization from 1/10 CH_2Cl_2/*n*-hexane. Spectroscopic data of **1** are as follows. IR (KBr, cm^{-1}) υ (CN) 1630 (m); υ (CS) 1481 (m), 1437 (m); υ (PF_6) 839 (vs). ^1H NMR (500 MHz, $CDCl_3$, 298 K): δ 2.45, 3.61 (s, 6H, NCH_3), 7.24-7.73 (m, 30H, Ph). $^{31}P\{^1H\}$ NMR (202 MHz, $CDCl_3$, 298 K): δ 23.2, 34.8 (d, PPh_3, $^2J_{P-P}$ = 40.7), -144.0 (sep, PF_6,

J_{P-F} = 708.6). $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$, 298 K): δ 45.9, 53.9 (s, NCH_3), 128.8-134.0 (m, C of Ph), 212.1 (d, CS, $^2J_{P-C}$ = 6.7). MS (FAB, NBA, *m/z*): 718 [M^+ - PF_6], 630 [M^+ - PF_6 - $CSNMe_2$], 456 [M^+ - PF_6- PPh_3]. Anal. Calcd. for $C_{39}H_{36}F_6NP_3SPd$: C, 54.21; H, 4.20; N, 1.62%. Found: C, 54.18; H, 4.46; N, 1.51.

Preparation of [Pd(PPh_3)(η^1-$SCNMe_2$){η^2-$S_2P(OEt)_2$}], 2

MeOH (20 mL) was added to a flask (100 mL) containing $(EtO)_2PS_2NH_4$ (0.203 g, 1.0 mmol) and **1** (0.863 g, 1.0 mmol). The solution was stirred for 10 min, and the yellow-orange solids **2** were formed which were isolated by filtration (G4), washed with *n*-hexane (2 × 10 mL) and subsequently dried under vacuum yielding [Pd(PPh_3)(η^1-$SCNMe_2$){η^2-$S_2P(OEt)_2$}], **2** (0.48 g, 75%). Further purification was accomplished by recrystallization from 1/10 CH_2Cl_2/*n*-hexane. Spectroscopic data of **2** are as follows. IR (KBr, cm^{-1}) υ (CS) 1480 (m), 1430 (m). ^1H NMR (500 MHz, $CDCl_3$, 298 K): δ 1.37 (t, 6H, OCH_2CH_3, J_{H-H} = 6.95), 2.93, 3.30 (s, 6H, NCH_3), 4.24 (q, 4H, OCH_2CH_3, J_{H-H} = 6.95), 7.24-7.59 (m, 15H, Ph). $^{31}P\{^1H\}$ NMR (202 MHz, $CDCl_3$, 298 K): δ 23.9 (s, PPh_3), 100.4 (s, PS_2). $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$, 298 K): δ 15.7 (s, OCH_2CH_3), 41.8, 47.1 (s, NCH_3), 64.2, 64.5 (s, OCH_2CH_3), 128.2-134.4 (m, C of Ph), 225.1 (s, NCS). MS (FAB, NBA, *m/z*): 642 [M^+], 553 [M^+ - $CSNMe_2$]. Anal. Calcd. for $C_{25}H_{31}NO_2P_2S_3Pd$: C, 46.76; H, 4.87; N, 2.18%. Found: C, 46.52; H, 4.66; N, 2.31.

Preparation of [Pd(PPh_3)(η^1-$SCNMe_2$)(η^2-S_2CNEt_2)], 3 and *trans*-[Pd(PPh_3)$_2$(η^1-$SCNMe_2$)(η^1-SNC_5H_4)], 4

Complex [Pd(PPh_3)(η^1-$SCNMe_2$)(η^2-S_2CNEt_2)], **3** and *trans*-[Pd(PPh_3)$_2$(η^1-$SCNMe_2$)(η^1-SNC_5H_4)], **4** were synthesized using the same procedure as that used in the synthesis of **2** by employing **1** and Et_2NCS_2Na and pySK, respectively. The yields are 69% for **3** and 86% for **4**.

Spectroscopic data of **3** are as follows. IR (KBr, cm^{-1}) υ (CN) 1516 (m), υ (CS) 1478 (m), 1435 (m). ^1H NMR (500 MHz, $CDCl_3$, 298 K): δ 1.23 (t, 6H, NCH_2CH_3, J_{H-H} = 6.82), 2.93, 3.31 (s, 6H, NCH_3), 3.67 (q, 4H, NCH_2CH_3, J_{H-H} = 6.82), 7.24-7.63 (m, 15H, Ph). $^{31}P\{^1H\}$ NMR (202 MHz, $CDCl_3$, 298 K): δ 25.3 (s, PPh_3). $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$, 298 K): δ 12.4 (s, NCH_2CH_3), 40.7, 47.2 (s, NCH_3), 43.8, 44.4 (s, NCH_2CH_3), 128.2-134.3 (m, C of Ph), 208.7 (s, NCS_2), 233.8 (s, NCS). MS (FAB, NBA, *m/z*): 605 [M^+], 516 [M^+ - $CSNMe_2$]. Anal. Calcd. for $C_{26}H_{31}N_2PS_3Pd$: C, 51.60; H, 5.16; N, 4.63%. Found: C, 51.82; H, 4.96; N, 4.41.

Spectroscopic data of **4** are as follows. IR (KBr, cm^{-1}) υ (CN) 1575 (m), υ (CS) 1483 (m), 1438 (m). ^1H NMR (500 MHz, $CDCl_3$, 298 K): δ 2.56 (s, 6H, NCH_3), 7.19-7.75 (m, 15H, Ph). $^{31}P\{^1H\}$ NMR (202 MHz, $CDCl_3$, 298 K): δ 20.9 (s, PPh_3). $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$, 298 K): δ 46.6 (s, NCH_3), 115.9 (s, 5-C of pyS), 128.0-139.2 (m, C of Ph). MS (FAB, NBA, *m/z*): 718 [M^+ - pyS], 456 [M^+ - pyS - PPh_3]. Anal. Calcd. for $C_{44}H_{40}N_2P_2S_2Pd$: C, 63.72; H, 4.86; N, 3.38%. Found: C, 64.08; H, 4.56; N, 3.41.

Preparation of [Pd(PPh_3)(η^1-$SCNMe_2$)(η^2-dppm)][PF_6], 5

CH_2Cl_2 (20 mL) was added to a flask (100 mL) containing **1** (0.863 g, 1.0 mmol) and dppm (0.384 g, 1.0 mmol). The solution was stirred for 10 min at room temperature. *n*-Hexane (15 mL) was added to the solution and a yellow precipitate was formed. The precipitate was collected by filtration (G4), washed with *n*-hexane (2 × 10 mL) and then dried in vacuo yielding 0.77 g (78%) of **5**. Spectroscopic data of **5** are as follows. IR (KBr, cm^{-1}) υ (CN) 1633 (m); υ (CS) 1485 (m), 1438 (m); υ (PF_6) 841 (vs). ^1H NMR (500 MHz, $CDCl_3$, 298 K): δ 2.15, 3.73 (s, 6H, NCH_3), 2.68 (t, 2H, PCH_2, J_{P-H} = 5.38), 7.00-7.92 (m, 35H, Ph). $^{31}P\{^1H\}$ NMR (202 MHz, $CDCl_3$, 298 K): δ 4.51, 11.4 (dd, PPh_2, $^2J_{P-P}$ = 25.4, 24.2), 29.6 (dd, PPh_3, J_{P-P} = 25.4, 24.2). $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$, 298 K): δ 30.9 (m, CH_2), 34.3, 45.2 (s, NCH_3), 128.4-137.1 (m, C of Ph). MS (FAB, NBA, *m/z*): 578 [M^+ - PPh_3]. Anal. Calcd. for $C_{46}H_{43}F_6NP_4SPd$: C, 56.02; H, 4.40; N, 1.42%. Found: C, 56.05; H, 4.46; N, 1.51.

Single-Crystal X-ray Diffraction Analysis of 1

A single crystal of **1** suitable for X-ray diffraction analysis was grown by recrystallization from 20:1 *n*-hexane/CH_2Cl_2. The diffraction data were collected at room temperature on an Enraf-Nonius CAD4 diffractometer equipped with graphite-monochromated Mo Kα (λ = 0.71073 Å) radiation. The raw intensity data were converted to structure factor amplitudes and their esd's after correction for scan speed, background, Lorentz, and polarization effects. An empirical absorption correction, based on the azimuthal scan data, was applied to the data. Crystallographic computations were carried out on a Microvax III computer using the NRCC-SDP-VAX structure determination package.[6]

A suitable single crystal of **1** was mounted on the top of a glass fiber with glue. Initial lattice parameters were determined from 24 accurately centered reflections with θ values in the range from 1.44 to 27.50°. Cell constants and other pertinent data were collected and are recorded in Table 1. Reflection data were collected using the θ/2θ scan method. Three

Table 1. Crystal Data and Refinement Details for Complex **1**·CH_2Cl_2

	1·CH_2Cl_2
chemical formula	$C_{40}H_{38}Cl_2F_6NP_3SPd$
formula weight	948.98
crystal system	monoclinic
space group	$P2_1/c$
a, Å	9.8101(1)
b, Å	23.5315(3)
c, Å	18.2112(2)
α, deg	90
β, deg	103.2337(5)
γ, deg	90
V, $Å^3$	4092.35(8)
Z	4
ρ_{calcd}, g cm^{-3}	1.540
μ, (Mo Kα), mm^{-1}	0.810
λ, Å	0.71073
T, K	150(1)
θ range, deg	1.44-27.50
Independent rflns	9389
No. of variables	498
R[a]	0.064
R_w[b]	0.148
S[c]	1.038

[a] $R = \Sigma||Fo| - |Fc||/\Sigma|Fo|$. [b] $Rw = [\Sigma w(|Fo| - |Fc|)^2]^{1/2}$; $w = 1/s^2(|Fo|)$. [c] Quality-of-fit = $[\Sigma w(|Fo| - |Fc|)^2/(N_{observed} - N_{parameters})]^{1/2}$.

check reflections were measured every 30 min throughout the data collection and showed no apparent decay. The merging of equivalent and duplicate reflections gave a total of 27983 unique measured data in which 9389 reflections with I > 2σ(I) were considered observed. The structure was first solved by using the heavy-atom method (Patterson synthesis), which revealed the positions of metal atoms. The remaining atoms were found in a series of alternating difference Fourier maps and least-squares refinements. The quantity minimized by the least-squares program was $\omega(|F_o|-|F_c|)^2$, where ω is the weight of a given operation. The analytical forms of the scattering factor tables for the neutral atoms were used.[7] The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included in the structure factor calculations in their expected positions on the basis of idealized bonding geometry but were not refined in least squares. All hydrogens were assigned isotropic thermal parameters 1-2 $Å^2$ larger then the equivalent Biso value of the atom to which they were bonded. The final residuals of this refinement were $R = 0.064$ and $Rw = 0.148$. Selected bond distances and angles are listed in Table 2. Tables of thermal parameters and selected final atomic coordinates are given in the Supporting Information.

Table 2. Selected Interatomic Distances (Å) and Angles (deg) for **1**

bond lengths		bond angles	
Pd(1)-C(1)	2.003(6)	P(1)-Pd(1)-P(2)	103.66(5)
Pd(1)-S(1)	2.3243(15)	C(1)-Pd(1)-S(1)	44.54(16)
Pd(1)-P(1)	2.3125(15)	S(1)-Pd(1)-P(2)	104.92(5)
Pd(1)-P(2)	2.3660(16)	C(1)-Pd(1)-P(1)	106.82(16)
S(1)-C(1)	1.667(6)	C(1)-S(1)-Pd(1)	57.4(2)
C(1)-N(1)	1.303(7)	S(1)-C(1)-Pd(1)	78.0(2)
N(1)-C(2)	1.468(7)	S(1)-C(1)-N(1)	131.5(4)
N(1)-C(3)	1.466(7)	C(1)-N(1)-C(2)	120.8(5)

Table 3. Atomic Coordinates and Equivalent Isotropic Displacement Coefficients for Important Atoms of **1**

Atom	x	y	z	B_{eq}
Pd(1)	6527(1)	-5(1)	1637(1)	23(1)
S(1)	7067(2)	111(1)	468(1)	32(1)
P(1)	5379(1)	275(1)	2553(1)	25(1)
P(2)	7904(2)	-785(1)	2195(1)	26(1)
N(1)	5406(5)	1026(2)	513(2)	27(1)
C(1)	6028(5)	559(2)	794(3)	26(1)
C(2)	5513(6)	1229(3)	-234(3)	35(2)
C(3)	4618(6)	1391(3)	921(3)	34(2)
C(4)	6212(5)	887(2)	3070(3)	27(1)
C(10)	3566(5)	473(2)	2139(3)	26(1)
C(16)	5209(6)	-238(3)	3277(3)	30(1)
C(22)	7055(6)	-1451(3)	2350(3)	31(1)
C(28)	9081(5)	-593(3)	3086(3)	29(1)
C(34)	9113(6)	-1022(3)	1633(3)	29(1)

E.S.Ds. refer to the last digit printed.

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