Synthesis of Two Isomers of (Diphenylphosphino)indene and Their Platinum(II) Complexes

Kathleen A. Fallis, Gordon K. Anderson,* and Nigam P. Rath

Department of Chemistry, University of Missouri—St. Louis, 8001 Natural Bridge Road, St. Louis, Missouri 63121

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Deprotonation of indene followed by addition of Ph_2PCl yields one isomer of $Ph_2PC_9H_7$ (**1a**) which, on standing, converts to a second (**1b**). The crystal structure of **1b** has been determined and reveals that the diphenylphosphino group is attached to an sp^2 carbon of the five-membered ring. The compound crystallizes in the monoclinic space group $P2_1/c$ with a = 8.214 (2) Å, b = 16.651 (7) Å, c = 11.970 (4) Å, β = 93.37 (2)°, V = 1634.3 (10) Å^3, and Z = 4. Least squares refinement converged at R = 0.0456 and R_w = 0.0415 based on 3280 reflections with $F > 4.0\sigma(F)$. A series of platinum complexes of the type $[PtCl_xMe_yL_2]$ ($x + y = 2$) has been isolated for each isomer. In the complexes of **1a** the presence of a chiral carbon in the $Ph_2PC_9H_7$ ligands results in the formation of two pairs of diastereomers, which could be distinguished by NMR spectroscopy.

Introduction

There has been considerable interest recently in the use of the (diphenylphosphino)cyclopentadienyl (dppc) ligand for the construction of heteronuclear[1] and homonuclear[2] bimetallic complexes. Our own work has included the synthesis of zirconium–platinum species[3] and, more recently, studies of dppc-bridged diplatinum systems.[4] As an extension of this work we are studying the related (diphenylphosphino)indenyl ligand, prepared by deprotonation of (diphenylphosphino)indene, $Ph_2PC_9H_7$. In the process we have investigated the chemistry of $Ph_2PC_9H_7$, which exists in two isomeric forms.

In this paper we report the preparation and interconversion of the two isomers of $Ph_2PC_9H_7$ and the crystal structure of the thermodynamically more stable form. We have isolated a series of platinum complexes of each isomer. Whereas platinum complexes containing mutually cis $Ph_2PC_5H_5$ ligands undergo a Diels–Alder coupling of the cyclopentadienyl groups,[5] no such reactions take place with mutually cis $Ph_2PC_9H_7$ ligands.

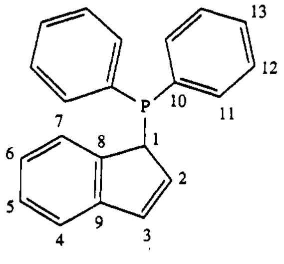
Results and Discussion

Table I. ^{1}H, ^{13}C{^{1}H}, and ^{31}P{^{1}H} NMR Data for **1a** and **1b**[a]

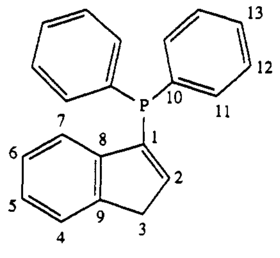
		1a[b]	**1b**[c]
^{1}H NMR	H_1	4.6 (br s)	
	H_2	6.5 (ddd) (5.4, 3.4, 1.7)	6.3 (dt) (3.3, 2.0)
	H_3	6.9 (br d) (5.4)	3.5 (dd) (3.4, 2.1)
^{13}C NMR	C_1	48.6 (d) (33.5)	145.9 (d) (19.9)
	C_2	132.1 (d) (5.2)	142.0 (d) (5.5)
	C_3	135.0 (d) (3.8)	40.2 (d) (5.7)
	C_4, C_5, C_6	121.5, 124.6, 126.6	124.1, 125.3, 126.4
	C_7	124.0 (d) (4.8)	121.5 (d) (4.8)
	C_8	144.0 (d) (9.1)	141.9 (d) (12.8)
	C_9	144.6 (d) (1.5)	144.8 (d) (4.9)
	C_{10}	136.8 (d) (10.9) 137.1 (d) (9.9)	136.0 (d) (8.8)
	C_{11}	133.0 (d) (19.8) 133.6 (d) (20.1)	134.2 (d) (20.0)
	C_{12}	128.0 (d) (6.8) 128.4 (d) (7.0)	128.8 (d) (7.3)
	C_{13}	128.9 129.2	129.4
^{31}P NMR		–4.3	–22.3

[a] Chemical shifts are in parts per million and are reported relative to residual solvent or external H_3PO_4, positive shifts representing deshielding. Coupling constants, J_{PC}, in hertz, are given in parentheses. [b] In $CDCl_3$ solution. [c] ^{1}H and ^{13}C spectra in CD_2Cl_2 solution; ^{31}P spectrum in $CDCl_3$ solution.

When an ether solution of indene was treated with 1 molar equiv of n-BuLi, deprotonation of the indene occurred, accompanied by an orange coloration of the solution. Subsequent addition of 1 molar equiv of chlorodiphenylphosphine resulted in the precipitation of TlCl and the formation of a bright yellow solution. After filtration and solvent removal, analysis by ^{31}P{^{1}H} NMR spectroscopy revealed the formation of a single phosphorus-containing compound. This was identified by ^{1}H and ^{13}C{^{1}H} NMR spectroscopy as **1a**. In particular, the ^{1}H NMR spectrum exhibits three signals of equal intensity for the hydrogens on the five-membered ring, and the sp^3 carbon (48.6 ppm) shows a large coupling to phosphorus, indicative of a one-bond coupling. Because C_1 is a chiral center, the



1a



1b

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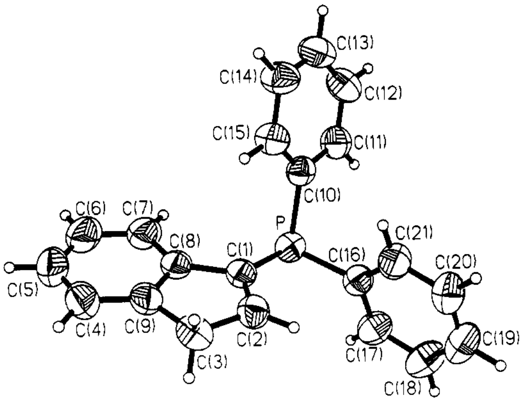

Figure 1. Projection view of the molecular structure of **1b**, showing the atom-labeling scheme.

Table II. Selected Bond Lengths (Å) and Angles (deg) for 1b

	Bond Lengths		
$P-C_1$	1.816 (2)	$P-C_{10}$	1.832 (2)
$P-C_{16}$	1.827 (2)	C_1-C_2	1.336 (3)
C_1-C_8	1.473 (2)	C_2-C_3	1.497 (3)
C_3-C_9	1.497 (3)	C_4-C_5	1.381 (4)
C_4-C_9	1.377 (3)	C_5-C_6	1.387 (3)
C_6-C_7	1.389 (4)	C_7-C_8	1.385 (3)
C_8-C_9	1.399 (3)		
	Bond Angles		
C_1-P-C_{10}	103.2 (1)	C_1-P-C_{16}	100.1 (1)
$C_{10}-P-C_{16}$	102.8 (1)	$P-C_1-C_2$	127.8 (1)
$P-C_1-C_8$	123.2 (1)	$C_2-C_1-C_8$	108.3 (2)
$C_1-C_2-C_3$	111.8 (2)	$C_2-C_3-C_9$	102.6 (2)
$C_3-C_9-C_4$	130.4 (2)	$C_9-C_4-C_5$	118.7 (2)
$C_4-C_5-C_6$	120.8 (3)	$C_5-C_6-C_7$	121.2 (2)
$C_6-C_7-C_8$	118.1 (2)	$C_1-C_8-C_7$	131.2 (2)
$C_1-C_8-C_9$	108.4 (2)	$C_7-C_8-C_9$	120.4 (2)
$C_3-C_9-C_8$	108.8 (2)	$C_4-C_9-C_8$	120.8 (2)

two phenyl groups attached to phosphorus are nonequivalent, and two sets of ^{13}C resonances are observed (Table I).

On standing in solution at ambient temperature for several days **1a** was converted to a second isomer **1b**. The isomerization may be followed readily by $^{31}P\{^1H\}$ NMR spectroscopy, the signal at −4.3 ppm being replaced by one at −22.3 ppm. The isomerization may be accelerated by passing the ether solution of **1a** through a short alumina column, when the conversion is quantitative within a few minutes. The thermodynamically more stable form, **1b**, has been characterized by NMR spectroscopy and by single-crystal X-ray diffraction. Its ^1H NMR spectrum exhibits two nonaromatic resonances in a 2:1 ratio at 3.5 and 6.3 ppm. The methylene carbon appears as a doublet at 40.2 ppm, but the coupling to phosphorus of only 5.7 Hz is indicative of a longer range coupling (Table I).

Light yellow, rhombic crystals of **1b** were obtained by slow evaporation of an ether solution. The compound crystallizes in the space group $P2_1/c$. The molecular structure of **1b** is shown in Figure 1, and selected bond lengths and angles are given in Table II. The C_1-C_2 distance of 1.336 Å is indicative of a double bond, whereas the C_2-C_3 and C_3-C_9 distances are identical at 1.497 Å. The $C_2-C_3-C_4$ angle is 102.6°, which is the smallest within the five-membered ring, as expected for an sp^3 carbon. The $P-C_1-C_2$ and $P-C_1-C_8$ angles are larger than the in-ring angles, but the geometry at C_1 is almost planar.

Table III. ^1H and $^{31}P\{^1H\}$ NMR Data for the Platinum Complexes Containing $Ph_2PC_9H_7$[a]

	^1H NMR	^{31}P NMR
3a		12.3, J_{PtP} 3714
		11.3, J_{PtP} 3682
4a[b]	−0.04 (t), J_{PH} 7, J_{PtH} 80	30.9, J_{PtP} 3066
	−0.07 (t), J_{PH} 7	30.6, J_{PtP} 3062
5a	0.90 (dd), J_{PH} 8, 7, J_{PtH} 67	25.1, J_{PtP} 1850
	0.86 (dd), J_{PH} 8, 7, J_{PtH} 70	25.0, J_{PtP} 1857
cis-**3b**		0.1, J_{PtP} 3607
trans-**3b**		22.3, J_{PtP} 2532
4b	−0.01 (t), J_{PH} 7, J_{PtH} 78	16.4, J_{PtP} 3102
5b	0.46 (dd), J_{PH} 9, 7, J_{PtH} 70	13.9, J_{PtP} 1836

[a] In $CDCl_3$ solution. Chemical shifts are in parts per million and are reported relative to residual solvent or external H_3PO_4, positive shifts representing deshielding. Coupling constants are in hertz. [b] J_{PtH} not observed for the second diastereomer.

When the solution of **1a** was allowed to remain in contact with the precipitate formed during its preparation, the isomerization of **1a** was slower than that observed for a filtered solution. In contrast, the rate of isomerization was enhanced greatly by the presence of alumina (neutral, acidic, or basic). The isomerization of **1a** to **1b** is the net result of transfer of a hydrogen from C_1 to C_3. We believe this most likely involves a protonation/deprotonation sequence, which could be catalyzed by either acid or base.

In contrast to the above, in the preparation of (diphenylphosphino)cyclopentadiene the isomer in which the PPh_2 group is attached to the sp^3 carbon was not detected. In the corresponding phosphine oxide and sulfide the two isomers in which the phosphino group is bonded to an sp^2 carbon were observed (in an approximately 85:15 ratio for the sulfide), and their interconversion was slow on the NMR time scale, but in $Ph_2PC_5H_5$ itself the two sp^2-bonded forms could not be distinguished.[6]

Solutions of **1a** or **1b** are oxygen-sensitive, but each phosphine could be converted cleanly to its sulfide, **2a** or **2b**, by stirring an ether solution with elemental sulfur under an inert atmosphere. The sulfide **2a** exhibits three nonaromatic resonances in its ^1H NMR spectrum, analogous to **1a**, whereas **2b** gives rise to the expected two signals in a 2:1 ratio. The ^{31}P resonances of **2a** and **2b** are shifted downfield by ca. 50 ppm relative to **1a** and **1b**, typical of oxidation to a phosphorus(V) compound. As in the case of the free phosphine, **2a** isomerized to **2b** on standing in solution.

Although **1a** converts slowly to **1b** in solution, a series of platinum complexes of **1a** could be prepared and isolated. These also react by isomerization of the phosphine ligand on standing in solution, so they were not crystallized in pure form but were characterized in solution by NMR spectroscopy. Addition of a freshly-prepared ether solution of **1a** to a CH_2Cl_2 solution of $[PtCl_2(cod)]$ resulted in formation of *cis*-$[PtCl_2(Ph_2PC_9H_7)_2]$ (**3a**), which was isolated as a yellow solid. Its $^{31}P\{^1H\}$ NMR spectrum consists of two singlets, each flanked by ^{195}Pt satellites, separated by 1 ppm and with almost identical $^1J_{PtP}$ coupling constants (Table III). Since C_1 in each $Ph_2PC_9H_7$ ligand is a chiral center, the platinum complex exists as two pairs of diastereomers which may be distinguished by NMR spectroscopy. The cis geometry follows from the magnitude of the $^1J_{PtP}$ coupling constants.[7]

Analogous reactions of **1a** with [PtClMe(cod)] or $[PtMe_2(cod)]$ produced *trans*-$[PtClMe(Ph_2PC_9H_7)_2]$ (**4a**) or *cis*-$[PtMe_2(Ph_2PC_9H_7)_2]$ (**5a**), respectively. In each case two singlets were observed in the $^{31}P\{^1H\}$ NMR spectrum, flanked by ^{195}Pt satellites, and two resonances were observed for the methyl hydrogens in the ^1H NMR spectrum. The equivalent phosphorus atoms in **4a** establish its trans

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geometry, whereas the doublet of doublets observed for the methyls and the small $^1J_{PtP}$ value indicate that the phosphines are mutually cis in **5a** (Table III).[7] When the reaction of **1a** with [PtClMe(cod)] was monitored by $^{31}P\{^1H\}$ NMR spectroscopy, it was found that *cis*-[PtClMe($Ph_2PC_9H_7$)$_2$] was formed initially, as evidenced by the appearance of two pairs of doublets with ^{195}Pt satellites (δ(P) 15.2 (d), $^1J_{PtP}$ 4462 Hz, 26.1 (d), $^1J_{PtP}$ 1752 Hz, $^2J_{PP}$ 11 Hz; δ(P) 15.6 (d), $^1J_{PtP}$ 4473 Hz, 27.0 (d), $^1J_{PtP}$ 1765 Hz, $^2J_{PP}$ 11 Hz). Isomerization to the trans isomer, **4a**, takes place on standing, and this is accelerated by the presence of excess $Ph_2PC_9H_7$.

The reactions of **1b** with [$PtCl_2$(cod)], [PtClMe(cod)], or [$PtMe_2$(cod)] proceeded analogously to give *cis*-[$PtCl_2$($Ph_2PC_9H_7$)$_2$] (*cis*-**3b**), *trans*-[PtClMe($Ph_2PC_9H_7$)$_2$] (**4b**) or *cis*-[$PtMe_2$($Ph_2PC_9H_7$)$_2$] (**5b**), respectively. The products were isolated as air-stable, analytically pure solids. For each complex the $^{31}P\{^1H\}$ NMR spectrum consists of a singlet with ^{195}Pt satellites, and the ^1H NMR spectra of the methyl complexes **4b** and **5b** each contain a single methyl resonance (Table III). In contrast to the reaction of **1b** with [$PtCl_2$(cod)], the reaction with [$PtCl_2$(NCPh)$_2$] produced *trans*-[$PtCl_2$($Ph_2PC_9H_7$)$_2$] (*trans*-**3b**). This was identified by its $^1J_{PtP}$ value of 2532 Hz, as compared with the value of 3607 Hz for *cis*-**3b**.[7] Since [$PtCl_2$(NCPh)$_2$] exists largely as the trans isomer,[8] this may simply represent a stereospecific displacement of the benzonitrile ligands by **1b**, whereas the reaction of [$PtCl_2$(cod)] yields the cis isomer.

Summary

We have shown that reaction of the indenyl anion with Ph_2PCl results in substitution at C_1, rather than C_2, and the initial product is that in which the PPh_2 group is attached to the sp^3 carbon (**1a**). This isomerizes to the thermodynamically more stable sp^2-substituted isomer (**1b**), which has been characterized by X-ray crystallography. Each isomer has been converted to its sulfide, and a series of platinum complexes of each isomer has been prepared. Since C_1 in **1a** is a chiral center, the bis(phosphine)platinum complexes of **1a** exist as two pairs of diastereomers, which can be distinguished by NMR spectroscopy.

Experimental Section

NMR spectra were recorded on a Varian XL-300 spectrometer, operating in the FT mode. ^1H and ^{13}C chemical shifts are relative to the residual solvent resonance, and ^{31}P shifts are relative to external 85% H_3PO_4, positive shifts representing deshielding. Microanalyses were performed by Atlantic Microlab, Inc., Norcross, GA. The compounds [$PtCl_2$(cod)], [$PtCl_2$(NCPh)$_2$], [$PtMe_2$(cod)], and [PtClMe(cod)] were prepared by established methods.[8-10] *n*-BuLi and Ph_2PCl were purchased from Aldrich and used without purification. Indene and all solvents were freshly distilled prior to use. The $Ph_2PC_9H_7$ ligands were prepared under an atmosphere of dry argon in a Vacuum Atmospheres controlled-atmosphere box. The platinum complexes were prepared by mixing a solution of the ligand with the appropriate precursor in the drybox, before removing the flask for workup.

Preparation of $Ph_2PC_9H_7$ (1a). Indene (0.20 mL, 1.72 mmol) was dissolved in diethyl ether (10 mL). While the solution was stirred vigorously, *n*-BuLi (1.06 mL, 1.6 M) was added over a period of 2 min. The solution turned orange. Slow addition of Ph_2PCl (0.30 mL, 1.67 mmol) produced a white precipitate of LiCl and a yellow solution. After filtration, $^{31}P\{^1H\}$ NMR analysis indicated complete conversion. The product was isolated by solvent removal, or its solution was used directly.

Table IV. Crystallographic Data for 1b

empirical formula	$C_{21}H_{17}P$
mol wt	300.32
color, habit	light yellow, rhombic
space group	$P2_1/c$
a, Å	8.214 (2)
b, Å	16.651 (7)
c, Å	11.970 (4)
β, deg	93.37 (2)
V, Å^3	1634.3 (10)
Z	4
D(calcd), Mg/m^3	1.221
temp, K	298
λ(Mo Kα radiation, graphite monochromated), Å	0.71073
crystal dimensions, mm	0.6 × 0.5 × 0.2
abs coeff, mm^{-1}	0.157
2θ range, deg	3.5–60.0
scan speed, deg/min	variable; 3.97–14.65
scan range (ω), deg	0.60 plus Kα separation
no. of ind reflns	4790
no. of obsd reflns	3280 ($F > 4.0\sigma(F)$)
no. of params refined	267
R	0.0456
R_w	0.0415
goodness of fit	0.51

Preparation of $Ph_2PC_9H_7$ (1b). An ether solution of **1a** was passed down a short column of alumina (8 cm, Brockman Activity 1, 80–200 mesh). Conversion to **1b** was quantitative, as determined by $^{31}P\{^1H\}$ NMR spectroscopy. Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of an ether solution.

Preparation of $Ph_2P(S)C_9H_7$ (2a). Powdered sulfur (0.38 g) was placed in a round-bottomed flask in the drybox. To this was added a solution of **1a** (4.0 mL, 0.15 M, 0.60 mmol). The mixture was stirred for 15 min and then filtered. The filtrate was evaporated to dryness, and the oily residue was dissolved in acetone. Removal of the acetone gave the product as a tan solid (0.18 g, 88%). ^1H NMR: δ 4.88 (d) (J_{PH} 24 Hz), 6.5 (m), 6.9 (m). $^{31}P\{^1H\}$ NMR: δ 45.1.

Preparation of $Ph_2P(S)C_9H_7$ (2b). A solution of **1b** (2.6 mL, 0.15 M, 0.39 mmol) was added to powdered sulfur (0.44 g). After stirring for 15 min, the mixture was filtered. The filtrate was reduced in volume, and pentane was added to precipitate the product as a yellow solid (0.11 g, 87%). Anal. Calcd for $C_{21}H_{17}PS$: C, 75.87; H, 5.15. Found: C, 75.60; H, 5.20. ^1H NMR: δ 3.6 (m), 6.64 (dt) (J_{PH} 11, J_{HH} 3.5 Hz). $^{31}P\{^1H\}$ NMR: δ 32.1.

Preparation of *cis*-[$PtCl_2$($Ph_2PC_9H_7$)$_2$] (3a). To a CH_2Cl_2 solution (35 mL) of [$PtCl_2$(cod)] (0.17 g, 0.45 mmol) was added an ether solution of **1a** (7.5 mL, 0.15 M, 1.12 mmol). After 30 min the solvent was removed. The resulting yellow solid was washed with pentane to remove excess phosphine and cyclooctadiene, leaving the product as a yellow solid (0.32 g, 82%).

Preparation of *trans*-[PtClMe($Ph_2PC_9H_7$)$_2$] (4a). This complex was generated as above from [PtClMe(cod)] (0.13 g, 0.37 mmol) and **1a** (6.2 mL, 0.15 M, 0.93 mmol) and isolated as a tan solid (0.30 g, 94%).

Preparation of *cis*-[$PtMe_2$($Ph_2PC_9H_7$)$_2$] (5a). This was prepared from [$PtMe_2$(cod)] (0.09 g, 0.27 mmol) and **1a** (4.5 mL, 0.15 M, 0.67 mmol) and obtained as a brown solid (0.20 g, 88%).

Preparation of *cis*-[$PtCl_2$($Ph_2PC_9H_7$)$_2$] (*cis*-3b). To a CH_2Cl_2 solution (35 mL) of [$PtCl_2$(cod)] (0.20 g, 0.55 mmol) was added a solution of **1b** (9.1 mL, 0.15 M, 1.36 mmol). The mixture was stirred for 30 min, and then the solvent was removed by rotary evaporation. The remaining tan solid was washed with pentane and dried in vacuo (0.42 g, 89%). Anal. Calcd for $C_{42}H_{34}Cl_2P_2Pt$: C, 58.20; H, 3.95. Found: C, 57.42; H, 3.99.

Preparation of *trans*-[PtClMe($Ph_2PC_9H_7$)$_2$] (4b). This compound was prepared as above from [PtClMe(cod)] (0.09 g, 0.26 mmol) and **1b** (4.4 mL, 0.15 M, 0.66 mmol) and obtained as a white solid (0.19 g, 85%). Anal. Calcd for $C_{43}H_{37}ClP_2Pt$: C, 61.03; H, 4.41. Found: C, 60.36; H, 4.44.

Preparation of *cis*-[$PtMe_2$($Ph_2PC_9H_7$)$_2$] (5b). This was prepared from [$PtMe_2$(cod)] (0.11 g, 0.32 mmol) and a solution

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Table V. Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Coefficients ($Å^2 \times 10^3$)

	x	y	x	U(eq)
P	4575 (1)	2596 (1)	824 (1)	51 (1)
C_1	4868 (2)	3373 (1)	−210 (1)	48 (1)
C_2	6276 (2)	3655 (1)	−556 (2)	55 (1)
C_3	6021 (3)	4372 (1)	−1299 (2)	61 (1)
C_4	3241 (3)	5072 (1)	−1832 (2)	74 (1)
C_5	1582 (4)	5049 (2)	−1701 (3)	89 (1)
C_6	908 (3)	4454 (2)	−1078 (3)	87 (1)
C_7	1873 (3)	3864 (1)	−552 (2)	68 (1)
C_8	3537 (2)	3887 (1)	−677 (2)	52 (1)
C_9	4212 (2)	4488 (1)	−1325 (2)	56 (1)
C_{10}	3792 (2)	1749 (1)	−23 (1)	48 (1)
C_{11}	3480 (3)	1042 (1)	545 (2)	62 (1)
C_{12}	2802 (3)	388 (1)	−12 (2)	73 (1)
C_{13}	2400 (3)	428 (1)	−1143 (2)	71 (1)
C_{14}	2697 (3)	1121 (1)	−1714 (2)	69 (1)
C_{15}	3392 (3)	1780 (1)	−1160 (2)	58 (1)
C_{16}	6693 (2)	2309 (1)	1164 (1)	51 (1)
C_{17}	7419 (3)	2591 (1)	2161 (2)	65 (1)
C_{18}	9061 (3)	2442 (2)	2440 (2)	81 (1)
C_{19}	9968 (3)	2007 (2)	1737 (2)	80 (1)
C_{20}	9265 (3)	1714 (2)	747 (2)	74 (1)
C_{21}	7628 (3)	1861 (1)	463 (2)	63 (1)

of **1b** (5.3 mL, 0.15 M, 0.79 mmol) and isolated as a yellow solid (0.225 g, 86%). Anal. Calcd for $C_{44}H_{40}P_2Pt$: C, 63.99; H, 4.88. Found: C, 63.75; H, 4.93.

Preparation of *trans*-[$PtCl_2(Ph_2PC_9H_7)_2$] (*trans*-3b). To a CH_2Cl_2 solution (20 mL) of [$PtCl_2(NCPh)_2$] (0.24 g, 0.50 mmol) was added an ether solution of **1b** (8.3 mL, 0.15 M, 1.24 mmol). After stirring for 15 min, the solvent was removed. The resulting yellow solid was washed with pentane and dried in vacuo (0.36 g, 83%). Anal. Calcd for $C_{42}H_{34}Cl_2P_2Pt$: C, 58.20; H, 3.95. Found: C, 58.07; H, 3.97.

X-ray Structure Determination. A single crystal of **1b** was mounted on a glass fiber in random orientation. Preliminary examination was carried out with Mo Kα radiation using a Siemens R3 automated four-circle diffractometer. Final cell parameters and orientation matrices were obtained by least squares refinement of 25 automatically centered reflections ($20° < 2\theta < 25°$). Axial photographs of the three axes were taken to confirm cell lengths and the lattice symmetry. ω-scans of representative reflections indicated acceptable crystal quality.

Data were collected using the θ–2θ scan technique, and the intensities of three standard reflections were measured every 50 reflections. As no significant variation in intensities of the standard reflections was observed during data collection, no decay correction was applied. No absorption correction was applied to the data.

Data reduction and structure solution was achieved using the SHELXTL PLUS structure solution software package.[11] The structure was solved by direct methods in the space group $P2_1/c$ and was refined successfully in this space group. The remaining atoms (hydrogen and non-hydrogen) were located from subsequent difference Fourier maps. Full-matrix least squares refinement was carried out by minimizing the function $w(F_o - F_c)^2$. All non-hydrogen atoms were refined anisotropically, and the hydrogen atoms isotropically, to convergence ($R = 4.56\%$, $R_w = 4.15\%$).

The final difference Fourier map had a maximum electron density of 0.25 e/Å^3. Least squares planes calculated for the phenyl rings showed very small deviations from planarity. A summary of the crystal data, intensity collection parameters, and final structure refinement parameters are presented in Table IV. Refined positional parameters for the non-hydrogen atoms are listed in Table V. All calculations were performed on a VAX Station II GPX computer using SHELXTL PLUS software.

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Supplementary Material Available: Tables of bond lengths and bond angles, anisotropic displacement coefficients for non-hydrogen atoms, and hydrogen atom coordinates and isotropic displacement coefficients (2 pages); a listing of observed and calculated structure factors (18 pages). Ordering information is given on any current masthead page.

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(11) Sheldrick, G. M. Siemens Analytical X-ray Division, Madison, WI, 1989.