

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1992). Cell refinement: *MSC/AFC Diffractometer Control software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1992). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1986). Program(s) used to refine structure: *TEXSAN LS*. Software used to prepare material for publication: *TEXSAN FINISH*.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters (\AA^2)*

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
Au(1)	0.54801 (7)	0.35615 (5)	0.5251 (1)	0.0403
Au(2)	0.43693 (7)	0.32211 (5)	0.21977 (10)	0.0337
Cl(1)	0.6889 (5)	0.2949 (4)	0.4228 (8)	0.0626
Cl(2)	0.4269 (5)	0.4549 (3)	0.1866 (7)	0.0502
P(1)	0.4152 (5)	0.4157 (4)	0.6343 (6)	0.0376
P(2)	0.4450 (5)	0.1923 (3)	0.2333 (6)	0.0310
C(12)	0.377 (2)	0.372 (2)	0.801 (3)	0.0550
C(13)	0.296 (2)	0.415 (1)	0.542 (3)	0.0679
C(22)	0.459 (2)	0.154 (1)	0.405 (2)	0.0451
C(23)	0.561 (2)	0.155 (1)	0.152 (2)	0.0439
C(111)	0.442 (2)	0.516 (1)	0.677 (2)	0.0275
C(112)	0.362 (2)	0.568 (1)	0.712 (3)	0.0447
C(113)	0.384 (2)	0.642 (2)	0.747 (3)	0.0662
C(114)	0.487 (2)	0.670 (2)	0.745 (2)	0.0541
C(115)	0.568 (2)	0.620 (2)	0.709 (3)	0.0538
C(116)	0.545 (2)	0.544 (1)	0.675 (2)	0.0339
C(211)	0.334 (2)	0.144 (1)	0.159 (3)	0.0426
C(212)	0.329 (2)	0.060 (1)	0.165 (3)	0.0490
C(213)	0.246 (2)	0.025 (1)	0.093 (3)	0.0630
C(214)	0.174 (2)	0.065 (2)	0.027 (4)	0.0771
C(215)	0.182 (2)	0.147 (2)	0.018 (3)	0.0740
C(216)	0.262 (2)	0.184 (1)	0.078 (4)	0.0742

Table 2. *Geometric parameters (\AA , $^\circ$)*

Au(1)—Cl(1)	2.276 (6)	P(1)—C(13)	1.74 (2)
Au(1)—P(1)	2.214 (6)	P(1)—C(111)	1.77 (2)
Au(2)—Cl(2)	2.273 (6)	P(2)—C(22)	1.76 (2)
Au(2)—P(2)	2.205 (5)	P(2)—C(23)	1.77 (2)
P(1)—C(12)	1.81 (2)	P(2)—C(211)	1.77 (2)
Cl(1)—Au(1)—P(1)	177.2 (3)	C(13)—P(1)—C(111)	106 (1)
Cl(2)—Au(2)—P(2)	175.4 (2)	Au(2)—P(2)—C(22)	115.2 (7)
Au(1)—P(1)—C(12)	114.9 (9)	Au(2)—P(2)—C(23)	111.6 (8)
Au(1)—P(1)—C(13)	114.9 (9)	Au(2)—P(2)—C(211)	113.7 (8)
Au(1)—P(1)—C(111)	113.4 (8)	C(22)—P(2)—C(23)	100 (1)
C(12)—P(1)—C(13)	101 (1)	C(22)—P(2)—C(211)	106 (1)
C(12)—P(1)—C(111)	104 (1)	C(23)—P(2)—C(211)	108 (1)

[(Me₂Ph)PAuCl] was prepared according to the literature procedure (Al-Saady, McAuliffe, Parish & Sandbank, 1985). Scans of $(0.94 + 0.35 \tan \theta)^\circ$ were made at $32^\circ \text{ min}^{-1}$ in ω . The ratio of peak to background counting times was 2:1. The absolute configuration was determined by reversing the signs of the reflections (other hand: $R = 0.047$ and $wR = 0.050$).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71195 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1052]

References

Al-Saady, A. K., McAuliffe, C. A., Parish, R. V. & Sandbank, J. A. (1985). *Inorg. Synth.* **23**, 191–194.

Baenziger, N. C., Bennett, W. E. & Soboroff, D. M. (1976). *Acta Cryst.* **B32**, 962–963.
 Harker, C. S. W. & Tiekink, E. R. T. (1990). *Acta Cryst.* **C46**, 1546–1547.
 Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
 Molecular Structure Corporation (1992). *TEXSAN. Single Crystal Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
 Molecular Structure Corporation (1992). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
 Muir, J. A., Muir, M. M., Pulgar, L. B., Jones, P. G. & Sheldrick, G. M. (1985). *Acta Cryst.* **C41**, 1174–1176.
 Sheldrick, G. M. (1986). *SHELXS86. Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.
 Tiekink, E. R. T. (1989). *Acta Cryst.* **C45**, 1233–1234.
 Tolman, C. A. (1977). *Chem. Rev.* **77**, 313–348.
 Walker, N. & Stuart, D. (1983). *Acta Cryst.* **A39**, 158–166.

Acta Cryst. (1993). **C49**, 1603–1606

Structure of (η^5 -Cyclopentadienyl)bis-(dimethylphenylphosphine)palladium(II) Perchlorate

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Abstract

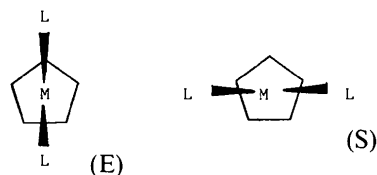
In the crystal structure of [Pd(η^5 -C₅H₅)(PMe₂Ph)₂]-ClO₄ the asymmetric unit contains two cations and two anions, one cation adopting a nearly eclipsed, and the other a staggered orientation of the C₅H₅ ring with respect to the perpendicular Pd(PMe₂Ph)₂ fragment. The coordination geometry around the metal atom is distorted square planar in both rotamers. The Pd—P distances are 2.250 (2)–2.253 (2) Å and the Pd—C distances vary from 2.252 (8)–2.343 (6) Å.

Comment

Interest in transition-metal complexes [M(η^5 -C₅H₅)L_x] stems from realization that binding to a ML_x fragment with metal-atom orbitals of less than cylindrical distribution causes the C₅H₅ ring to lose its D_{5h} symmetry.

Significant variations in the C—M and C—C bond lengths have been observed in the complexes of the

d^8 metal ions of types $[M(\eta^5\text{-C}_5\text{H}_5)\text{L}_2]$ and $[M(\eta^5\text{-C}_5\text{H}_5)\text{LL}']$. These complexes display eclipsed (E) or staggered (S) molecular structures, depending on orientation of the C_5H_5 ring with respect to the perpendicular ML_2 or MLL' fragment; the two



geometries are distinguished by different patterns of ring distortion (Byers & Dahl, 1980; Barker, Cameron, Kilner, Mahmood & Wallwork, 1991). The origin of these distortions is not yet fully understood (Albinati, Affolter & Pregosin, 1990).

Several palladium(II) complexes of this type have been characterized crystallographically and shown to adopt an eclipsed conformation. As part of our study of such complexes (Anderson, Cross, Manojlović-Muir, Muir & Rocamora, 1988; Anderson, Cross, Muir & Manojlović-Muir, 1989), we now report the crystal structure of the title compound in which eclipsed conformers of the $[\text{Pd}(\eta^5\text{-C}_5\text{H}_5)(\text{PMe}_2\text{Ph})_2]^+$ cation coexist with staggered ones in the ratio 1:1.

The compound was prepared by a variation of the method described by Roberts, Skelton, White & Wild (1982). A suspension of *trans*- $[\text{PdCl}_2(\text{PMe}_2\text{Ph})_2]$ in acetone was treated first with a solution of AgClO_4 and then with cyclopentadiene. The red precipitate was recrystallized several times from a dichloromethane/diethyl ether mixture to obtain crystals of a quality suitable for X-ray analysis. They were characterized by elemental analysis (found: C 45.3, H 4.9%; calculated for $\text{C}_{21}\text{H}_{27}\text{ClO}_4\text{P}_2\text{Pd}$: C 46.1, H 4.9%) and by NMR spectroscopy [^1H NMR: δ 1.61 ($J_{\text{P-H}} + J_{\text{P-H}} = 10.94$ Hz) CH_3 , δ 5.88 (t , 1.96 Hz) C_5H_5 ; ^{31}P NMR: δ 3.35 p.p.m.]. The methyl(phosphine) proton NMR signal is second order, indicative of a larger $J_{\text{P-P}}$ than usually found in *cis*-bisphosphine complexes of square-planar geometry (but not as high as expected for *trans* compounds).

The crystal structure of $[\text{Pd}(\eta^5\text{-C}_5\text{H}_5)(\text{PMe}_2\text{Ph})_2]\text{ClO}_4$ is built of asymmetric units each comprising two cations and two anions. The ClO_4^- ions may be subject to a slight disorder, as suggested by the relatively high displacement parameters of the Cl and O atoms. The geometry of the ClO_4^- ions shows no unexpected features.

The structures of the two symmetry independent cations, *A* and *B*, are shown in Fig. 1. In each cation, the dihedral angle between the PdP_2 and C_5H_5 planes (87.8 in *A* and 89.9° in *B*) and the P—Pd—P bond angle [95.7 (1) in *A* and 98.0 (1) $^\circ$ in *B*] approximate

to 90° . Thus, in both cations, the geometry around the metal centre can be described as distorted square planar, with two mutually *cis* coordination sites occupied by a C_5H_5 ligand. The distortions of the P—Pd—P angles are considerable and may reflect steric requirements of the phosphine ligands. The Pd—P bond lengths [2.250 (2)—2.253 (2) Å] are the same in both cations and they are in agreement with those [2.241 (3)—2.266 (3) Å] observed in $[\text{Pd}(\eta^5\text{-C}_5\text{H}_5)(\eta^1\text{-C}_5\text{H}_5)(\text{P}^i\text{Pr}_3)]$, $[\text{Pd}(\eta^5\text{-C}_5\text{H}_5)(\text{Ph}_2\text{PCH}_2\text{CH}_2\text{-PPh}_2)][\text{CF}_3\text{SO}_3]$ and $[\text{Pd}(\eta^5\text{-C}_5\text{H}_5)(\text{C}_6\text{H}_4\text{-2-N=NPh})(\text{PCy}_3)]$ (where Cy is cyclohexyl) (Werner, Kraus, Schubert, Ackerman & Hofmann, 1983; Bachechi, Lehmann & Venanzi, 1988; Anderson, Cross, Manojlović-Muir, Muir & Rocamora, 1988).

The main differences between cations *A* and *B* lie in their conformations and in the distortions of the C_5H_5 rings from the D_{5h} symmetry. The rotational orientation of the C_5H_5 ring with respect to the perpendicular $\text{Pd}(\text{PMe}_2\text{Ph})_2$ fragment is close to eclipsed in *A* and almost exactly staggered in *B* (Fig. 1). Furthermore, the phenyl substituents at the P atoms adopt an *anti* arrangement with respect to the PdP_2 plane in *A*, and a *syn* arrangement in *B*. In both cations the atomic displacement parameters indicate a considerable librational motion of the C_5H_5 ring (Fig. 1), suggesting a low barrier to ring rotation which is consistent with the presence of both eclipsed and staggered conformers in the crystal structure of this compound.

The Pd—C distances reveal significant distortions of the $\text{Pd}(\eta^5\text{-C}_5\text{H}_5)$ fragment from D_5 symmetry. In *A*, the Pd(1)—C(R15) distance [2.259 (7) Å] is shorter and the Pd(1)—C(R11) distance [2.343 (6) Å] is longer than the remaining three [2.301 (8)—2.317 (8) Å]. One short Pd—C(ring) distance appears to be a common feature of the crystallographically characterized $[\text{Pd}(\eta^5\text{-C}_5\text{H}_5)\text{L}_2]$ and $[\text{Pd}(\eta^5\text{-C}_5\text{H}_5)\text{LL}']$ complexes (Anderson, Cross, Muir & Manojlović-Muir, 1989; Albinati, Affolter & Pregosin, 1990) and this has been rationalized by a molecular-orbital treatment of the hypothetical complex $[\text{Pd}(\eta^5\text{-C}_5\text{H}_5)(\text{CH}_3)(\text{PH}_3)]^+$ (Werner, Kraus, Schubert, Ackerman & Hofmann, 1983). However, variations of the remaining four Pd—C(ring) distances observed in these complexes reveal a few different distortion patterns, which are not in accord with the theoretical predictions of Werner, Kraus, Schubert, Ackerman & Hofmann (1983). In the staggered cation *B*, two Pd—C distances [Pd(2)—C(R23) 2.252 (8) and Pd(2)—C(R21) 2.270 (6) Å] are shorter than the other three [2.324 (6)—2.341 (6) Å]. The same symmetrical pattern of the M—C(ring) bonds has been found previously in some staggered complexes of cobalt and rhodium, and predicted by a molecular-orbital treatment of the $[\text{Rh}(\eta^5\text{-C}_5\text{H}_5)(\text{CO})_2]$ complex (Lichtenberger, Blevins & Ortega, 1984).

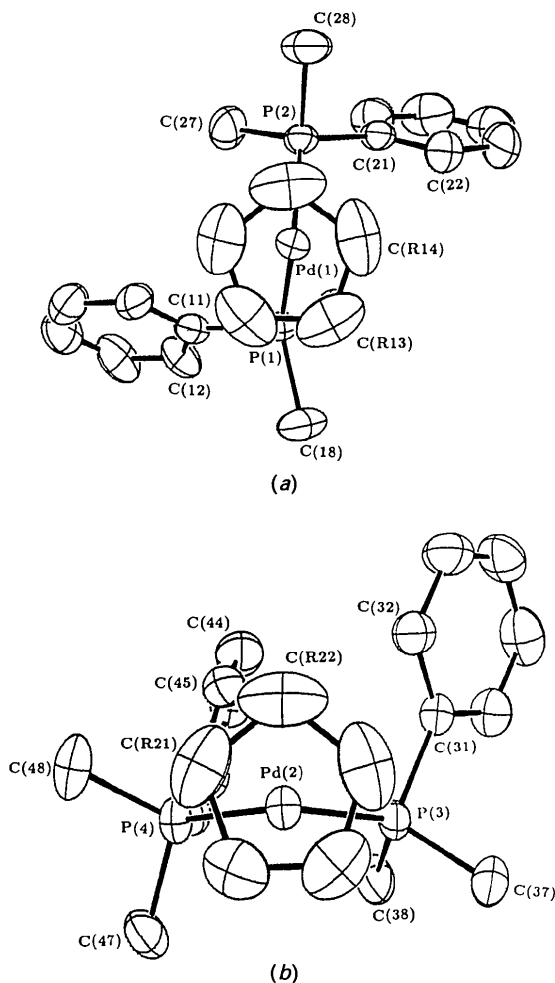


Fig. 1. The structures of two symmetry independent cations, illustrating (a) the eclipsed conformation of *A* and (b) the staggered conformation of *B*. The C atoms of the phenyl groups are numbered cyclically, C(*n*), C(*n* + 1), ..., C(*n* + 5) and only two are labelled for clarity. In the C₅H₅ rings, the atoms are also numbered cyclically, C(R*n*1), C(R*n*2), ..., C(R*n*5), with *n* = 1 in *A* and *n* = 2 in *B*. The atoms P(1) and C(17) in *A*, and C(41), C(42), C(43) and C(46) in *B* are obscured by the C₅H₅ rings.

The C—C ring distances also show significant variations. In *A*, one bond [C(R11)—C(R12) 1.303 (14) Å] is shorter than the other four [1.371 (12)–1.414 (13) Å], while in *B*, two adjacent bonds [C(R24)—C(R25) 1.341 (10) and C(R23)—C(R24) 1.346 (12) Å] are shorter than the remaining three [1.406 (12)–1.431 (12) Å]. Such distortions from the *D*_{5h} ring symmetry are different from those predicted for the eclipsed and staggered geometries (Werner, Kraus, Schubert, Ackerman & Hofmann, 1983; Lichtenberger, Blevins & Ortega, 1984) and those found in some other Pd(η^5 -C₅H₅) complexes. Indeed, some C—C bond sequences observed in other palladium(II) complexes are also in disagreement with theoretical predictions (Anderson,

Cross, Manojlović-Muir, Muir & Rocamora, 1988; Albinati, Affolter & Pregosin, 1990). It is therefore obvious that further studies of the [M(η^5 -C₅H₅)L₂] and [M(η^5 -C₅H₅)LL] complexes are needed before their structural properties can be fully understood.

Experimental

Crystal data

[Pd(C₅H₅)(C₈H₁₁P)₂]ClO₄
M_r = 547.3
 Triclinic
*P*1
a = 8.9982 (6) Å
b = 9.1024 (7) Å
c = 16.7740 (7) Å
 α = 88.285 (5)°
 β = 74.676 (5)°
 γ = 63.459 (6)°
V = 1179.2 (2) Å³
Z = 2

D_x = 1.541 Mg m⁻³
 Mo K α radiation
 λ = 0.71069 Å
 Cell parameters from 25 reflections
 θ = 14–17°
 μ = 1.047 mm⁻¹
T = 296 K
 Plate
 0.48 × 0.48 × 0.18 mm
 Red

Data collection

Enraf-Nonius CAD-4 diffractometer
 $\theta/2\theta$ scans
 Absorption correction: empirical; Walker & Stuart (1983)
 T_{\min} = 0.99, T_{\max} = 1.19
 5320 measured reflections
 5320 independent reflections

4845 observed reflections
 $[I \geq 3\sigma(I)]$
 θ_{\max} = 27°
 h = -11 → 11
 k = -11 → 11
 l = 0 → 21
 2 standard reflections
 frequency: 120 min
 intensity variation: <5%

Refinement

Refinement on *F*
 Final *R* = 0.0238
 wR = 0.0312
 S = 1.63
 4845 reflections
 521 parameters
 $w = 1/\sigma^2(|F_o|)$

$(\Delta/\sigma)_{\max}$ = 0.042
 $\Delta\rho_{\max}$ = 0.73 e Å⁻³
 $\Delta\rho_{\min}$ = -0.45 e Å⁻³
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

	$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$			
	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
Pd(1)	0.16929 (2)	0.07560 (2)	0.24628 (1)	0.037
Pd(2)	-0.16929	-0.07560	-0.24628	0.037
Cl(1)	-0.49127 (17)	-0.40570 (16)	0.63375 (10)	0.068
Cl(2)	0.55075 (14)	-0.52628 (15)	0.13592 (8)	0.053
P(1)	-0.06897 (12)	0.10756 (13)	0.34622 (7)	0.039
P(2)	0.35241 (13)	-0.03038 (14)	0.32557 (7)	0.043
P(3)	-0.21796 (13)	0.10677 (12)	-0.14286 (7)	0.039
P(4)	-0.15188 (15)	-0.28861 (12)	-0.17089 (7)	0.043
O(11)	-0.5669 (9)	-0.2382 (9)	0.6279 (7)	0.167
O(12)	-0.6001 (13)	-0.4689 (14)	0.6747 (6)	0.198
O(13)	-0.4679 (19)	-0.4509 (24)	0.5508 (8)	0.312
O(14)	-0.3252 (12)	-0.4787 (14)	0.6366 (9)	0.238
O(21)	0.6422 (9)	-0.6993 (6)	0.1338 (4)	0.115
O(22)	0.3822 (7)	-0.4706 (10)	0.1877 (4)	0.135
O(23)	0.6365 (8)	-0.4539 (8)	0.1653 (4)	0.129
O(24)	0.5436 (7)	-0.4868 (7)	0.0536 (3)	0.104

C(11)	-0.0864 (5)	-0.0837 (6)	0.3589 (3)	0.047	P(3)—Pd(2)—P(4)	98.0 (1)	P(3)—Pd(2)—C(R21)	158.7 (2)
C(12)	-0.1988 (7)	-0.1004 (8)	0.4283 (3)	0.067	P(3)—Pd(2)—C(R22)	124.3 (3)	P(3)—Pd(2)—C(R23)	99.9 (2)
C(13)	-0.2112 (10)	-0.2462 (10)	0.4366 (4)	0.092	P(3)—Pd(2)—C(R24)	108.3 (2)	P(3)—Pd(2)—C(R25)	138.9 (2)
C(14)	-0.1105 (10)	-0.3767 (8)	0.3743 (5)	0.092	P(4)—Pd(2)—C(R21)	102.0 (2)	P(4)—Pd(2)—C(R22)	125.1 (3)
C(15)	-0.0014 (8)	-0.3598 (7)	0.3058 (5)	0.083	P(4)—Pd(2)—C(R23)	160.6 (2)	P(4)—Pd(2)—C(R24)	142.7 (2)
C(16)	0.0129 (6)	-0.2145 (6)	0.2969 (4)	0.061	P(4)—Pd(2)—C(R25)	111.7 (2)	Pd(2)—P(3)—C(31)	113.4 (2)
C(17)	-0.0964 (7)	0.1846 (6)	0.4499 (3)	0.058	Pd(2)—P(3)—C(37)	110.6 (2)	Pd(2)—P(3)—C(38)	119.3 (2)
C(18)	-0.2667 (7)	0.2525 (8)	0.3234 (4)	0.070	C(31)—P(3)—C(37)	104.4 (3)	C(31)—P(3)—C(38)	105.6 (3)
C(21)	0.3223 (5)	0.1097 (6)	0.4099 (3)	0.048	C(37)—P(3)—C(38)	102.0 (3)	Pd(2)—P(4)—C(41)	116.8 (2)
C(22)	0.2918 (8)	0.2700 (7)	0.3932 (4)	0.071	Pd(2)—P(4)—C(47)	113.3 (2)	Pd(2)—P(4)—C(48)	111.3 (2)
C(23)	0.2776 (9)	0.3782 (8)	0.4543 (5)	0.089	C(41)—P(4)—C(47)	106.1 (3)	C(41)—P(4)—C(48)	103.8 (3)
C(24)	0.2863 (9)	0.3304 (11)	0.5323 (5)	0.093	C(47)—P(4)—C(48)	104.3 (3)	C(R22)—C(R21)—C(R25)	108.0 (7)
C(25)	0.3122 (10)	0.1723 (12)	0.5490 (4)	0.095	C(R21)—C(R22)—C(R23)	104.2 (7)	C(R22)—C(R23)—C(R24)	109.5 (7)
C(26)	0.3302 (8)	0.0623 (8)	0.4883 (4)	0.072	C(R23)—C(R24)—C(R25)	110.0 (7)	C(R21)—C(R25)—C(R24)	107.9 (6)
C(27)	0.3605 (8)	-0.2155 (7)	0.3703 (4)	0.072				
C(28)	0.5741 (7)	-0.0897 (8)	0.2640 (4)	0.072				
C(31)	-0.0240 (5)	0.1177 (5)	-0.1376 (3)	0.042				
C(32)	0.1285 (6)	0.0348 (6)	-0.2008 (3)	0.054				
C(33)	0.2756 (7)	0.0478 (7)	-0.1990 (4)	0.071				
C(34)	0.2720 (8)	0.1411 (8)	-0.1365 (4)	0.076				
C(35)	0.1211 (9)	0.2236 (8)	-0.0719 (4)	0.078				
C(36)	-0.0261 (7)	0.2112 (6)	-0.0731 (3)	0.057				
C(37)	-0.3626 (7)	0.3164 (6)	-0.1590 (4)	0.064				
C(38)	-0.3234 (7)	0.0901 (6)	-0.0378 (3)	0.062				
C(41)	-0.0481 (5)	-0.3168 (5)	-0.0884 (3)	0.042				
C(42)	-0.1242 (6)	-0.3328 (6)	-0.0073 (3)	0.055				
C(43)	-0.0407 (8)	-0.3511 (7)	0.0533 (3)	0.069				
C(44)	0.1177 (9)	-0.3530 (8)	0.0342 (4)	0.077				
C(45)	0.1965 (7)	-0.3397 (7)	-0.0465 (4)	0.073				
C(46)	0.1143 (6)	-0.3203 (6)	-0.1081 (3)	0.058				
C(47)	-0.3592 (7)	-0.2833 (7)	-0.1235 (4)	0.067				
C(48)	-0.0262 (9)	-0.4854 (6)	-0.2359 (4)	0.070				
C(R11)	0.2548 (13)	-0.0275 (9)	0.1067 (3)	0.091				
C(R12)	0.0931 (12)	0.0830 (12)	0.1247 (4)	0.108				
C(R13)	0.0760 (10)	0.2392 (10)	0.1450 (4)	0.088				
C(R14)	0.2416 (13)	0.2187 (10)	0.1387 (4)	0.103				
C(R15)	0.3507 (8)	0.0472 (12)	0.1190 (4)	0.092				
C(R21)	-0.0913 (10)	-0.1927 (8)	-0.3778 (3)	0.083				
C(R22)	-0.0013 (8)	-0.0990 (12)	-0.3815 (3)	0.092				
C(R23)	-0.1350 (12)	0.0666 (9)	-0.3560 (4)	0.100				
C(R24)	-0.2899 (9)	0.0714 (8)	-0.3481 (4)	0.078				
C(R25)	-0.2704 (8)	-0.0818 (9)	-0.3605 (3)	0.077				

Table 2. Selected bond lengths (Å) and angles (°)

Pd(1)—P(1)	2.250 (2)	Pd(1)—P(2)	2.253 (2)
Pd(1)—C(R11)	2.343 (6)	Pd(1)—C(R12)	2.307 (8)
Pd(1)—C(R13)	2.301 (8)	Pd(1)—C(R14)	2.317 (8)
Pd(1)—C(R15)	2.259 (7)	P(1)—C(11)	1.818 (5)
P(1)—C(17)	1.808 (5)	P(1)—C(18)	1.812 (6)
P(2)—C(21)	1.815 (5)	P(2)—C(27)	1.803 (7)
P(2)—C(28)	1.822 (6)	C(R11)—C(R12)	1.303 (14)
C(R11)—C(R15)	1.371 (12)	C(R12)—C(R13)	1.402 (13)
C(R13)—C(R14)	1.391 (13)	C(R14)—C(R15)	1.414 (13)
Pd(2)—P(3)	2.253 (1)	Pd(2)—P(4)	2.252 (2)
Pd(2)—C(R21)	2.270 (6)	Pd(2)—C(R22)	2.324 (6)
Pd(2)—C(R23)	2.252 (8)	Pd(2)—C(R24)	2.331 (7)
Pd(2)—C(R25)	2.341 (6)	P(3)—C(31)	1.817 (5)
P(3)—C(37)	1.829 (6)	P(3)—C(38)	1.805 (6)
P(4)—C(41)	1.816 (5)	P(4)—C(47)	1.804 (6)
P(4)—C(48)	1.825 (5)	C(R21)—C(R22)	1.406 (12)
C(R21)—C(R25)	1.421 (11)	C(R22)—C(R23)	1.431 (12)
C(R23)—C(R24)	1.346 (12)	C(R24)—C(R25)	1.341 (10)
P(1)—Pd(1)—P(2)	95.7 (1)	P(1)—Pd(1)—C(R11)	128.0 (3)
P(1)—Pd(1)—C(R12)	103.9 (3)	P(1)—Pd(1)—C(R13)	106.9 (2)
P(1)—Pd(1)—C(R14)	137.5 (3)	P(1)—Pd(1)—C(R15)	160.5 (2)
P(2)—Pd(1)—C(R11)	120.5 (3)	P(2)—Pd(1)—C(R12)	153.0 (3)
P(2)—Pd(1)—C(R13)	151.2 (3)	P(2)—Pd(1)—C(R14)	116.9 (3)
P(2)—Pd(1)—C(R15)	102.5 (2)	Pd(1)—P(1)—C(11)	112.4 (2)
Pd(1)—P(1)—C(17)	117.9 (2)	Pd(1)—P(1)—C(18)	112.4 (3)
C(11)—P(1)—C(17)	104.8 (3)	C(11)—P(1)—C(18)	105.2 (3)
C(17)—P(1)—C(18)	103.0 (3)	Pd(1)—P(2)—C(21)	116.0 (2)
Pd(1)—P(2)—C(27)	115.3 (3)	Pd(1)—P(2)—C(28)	110.5 (2)
C(21)—P(2)—C(27)	106.5 (3)	C(21)—P(2)—C(28)	102.7 (3)
C(27)—P(2)—C(28)	104.5 (3)	C(R12)—C(R11)—C(R15)	108.3 (8)
C(R11)—C(R12)—C(R13)	110.6 (9)	C(R12)—C(R13)—C(R14)	106.7 (8)
C(R13)—C(R14)—C(R15)	105.3 (8)	C(R11)—C(R15)—C(R14)	108.8 (8)

The diffraction pattern was compatible with the space groups $P1$ and $P\bar{1}$, but the Patterson function indicated $P1$ with two molecules per asymmetric unit. The structure was solved by Patterson and Fourier methods and refined by least squares using a two-large-blocks approximation to the normal matrix. The origin of the unit cell was defined by constraining the coordinates of the Pd(2) atom to $x_2 = -x_1$, $y_2 = -y_1$, $z_2 = -z_1$, where x_1 , y_1 and z_1 define the position of the Pd(1) atom. All non-H atoms were assigned anisotropic displacement parameters. The H atoms were included in the structural model in calculated positions (C—H = 0.96 Å) and allowed to ride on the C atoms to which they are bonded with $U(\text{H}) = 1.2 \times U_{\text{iso}}(\text{C})$. The absolute structure was determined by refining the polarity parameter (Rogers, 1981) to converge at $\eta = 0.95$ (5) and $(1 + |\eta|)/\sigma(\eta) = 36.4$. All calculations were performed with the GX program package (Mallinson & Muir, 1985) which includes a local modification of ORTEP (Johnson, 1971).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71160 (33 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1039]

References

- Albinati, A., Affolter, S. & Pregosin, P. S. (1990). *Organometallics*, **9**, 379–387.
- Anderson, G. K., Cross, R. J., Manojlović-Muir, Lj., Muir, K. W. & Rocamora, M. (1988). *Organometallics*, **7**, 1520–1525.
- Anderson, G. K., Cross, R. J., Muir, K. W. & Manojlović-Muir, Lj. (1989). *J. Organomet. Chem.* **362**, 225–235.
- Bachechi, F., Lehmann, R. & Venanzi, L. M. (1988). *J. Crystallogr. Spectrosc. Res.* **18**, 721–728.
- Barker, J., Cameron, N. D., Kilner, M., Mahmood, M. M. & Wallwork, S. C. (1991). *J. Chem. Soc. Dalton Trans.* pp. 3435–3445.
- Byers, R. L. & Dahl, L. F. (1980). *Inorg. Chem.* **19**, 277–284.
- Johnson, C. K. (1971). *ORTEP*. Report ORNL-3794, revised. Oak Ridge National Laboratory, Tennessee, USA.
- Lichtenberger, D. L., Blevins, C. H. & Ortega, P. B. (1984). *Organometallics*, **3**, 1614–1622.
- Mallinson, P. R. & Muir, K. W. (1985). *J. Appl. Cryst.* **18**, 51–53.
- Roberts, N. K., Skelton, B. W., White, A. N. & Wild, S. B. (1982). *J. Chem. Soc. Dalton Trans.* pp. 2093–2097.
- Rogers, D. (1981). *Acta Cryst.* **A37**, 734–741.
- Walker, N. & Stuart, D. (1983). *Acta Cryst.* **A39**, 158–166.
- Werner, H., Kraus, H.-J., Schubert, U., Ackerman, K. & Hofmann, P. (1983). *J. Organomet. Chem.* **250**, 517–536.