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Structure of *cis*-Bis(pentafluorophenyl)bis(triphenylphosphine)palladium(II)

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Abstract. [Pd{(C$_6$H$_5$)$_3$P}$_2$(C$_6$F$_5$)$_2$], $M_r = 965{\cdot}1$, orthorhombic, *Pbca*, $a = 18{\cdot}046$ (4), $b = 21{\cdot}189$ (9), $c = 21{\cdot}643$ (4) Å, $U = 8276$ (5) Å^3, $Z = 8$, $D_x = 1{\cdot}549$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 6{\cdot}05$ cm^{-1}, $F(000) = 3872$, $T = 295$ K, $R = 0{\cdot}053$ for 4923 observed reflections. The molecule has approximate C_2 symmetry, where the twofold axis is the bisector of the P(1)–Pd–P(2) angle. The geometry around the Pd atom is square-planar. The Pd, P(1), P(2), C(71) and C(81) atoms are essentially coplanar with a maximum atomic deviation of 0·35 Å from the least-squares plane. The planes of the C$_6$F$_5$ groups are almost perpendicular to the coordination plane. This result affords one of the fundamental structures in four-coordinated *cis*-diphosphine Pd^{II} complexes.

Experimental. Crystals of the title compound were grown as light yellow prisms from CH$_2$Cl$_2$/*n*-hexane solution. A well shaped crystal with approximate dimensions 0·25 × 0·25 × 0·35 mm was mounted on a Rigaku automated four-circle diffractometer. Unit-cell parameters were determined by least-squares refinement of 2θ values of 25 centered reflections with 2θ range 17·1–28·8°. Intensities were measured by the θ–2θ scan technique using graphite-monochromatized Mo $K\alpha$ radiation ($\lambda = 0{\cdot}71069$ Å). The scan rate was 8° min^{-1} in 2θ and the scan width was $\Delta(2\theta) = (2{\cdot}0 + 0{\cdot}70\tan\theta)°$. Background intensities were measured for 4 s at each end of a scan. Three standard reflections (800, 0,12,0, 0,0,10) were remeasured after every 56 reflections, no significant loss of these intensities being observed throughout data collection. Totals of 7488 independent reflections were collected with 2θ up to 50·5° [$(\sin\theta)/\lambda = 0{\cdot}600$ Å^{-1}] and index range $h = 0$ to 18, $k = 0$ to 21, $l = 0$ to 25. Corrections for Lorentz and polarization effects were applied to the intensity data, while no absorption or extinction correction was carried out.

The structure was solved by the heavy-atom method. All the non-H atoms were reasonably located on the

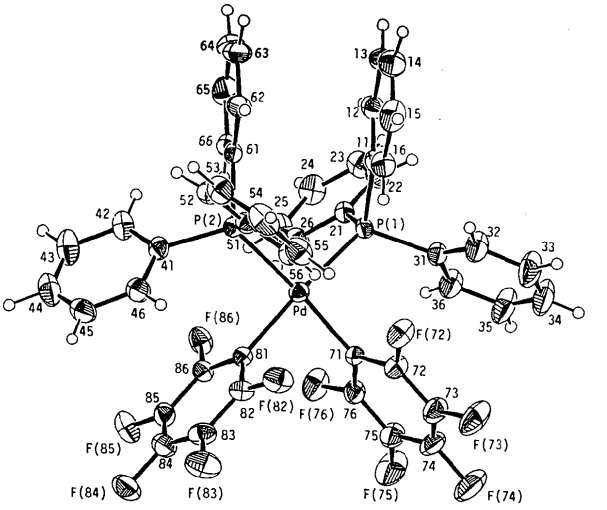

Fig. 1. *ORTEP* drawing (Johnson, 1976) of the molecular structure together with atomic numbering system. For carbon atoms only atomic numbers are shown for clarity. Non-H atoms are represented by thermal ellipsoids with 30% probability levels; H atoms are drawn by a sphere with $B = 1{\cdot}0$ Å^2.

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Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters of non-*H *atoms with e.s.d.'s in parentheses*

	x	y	z	B_{eq}*(Å²)
Pd	0·18593 (3)	0·11345 (2)	0·09145 (2)	3·2
P(1)	0·24822 (9)	0·07093 (8)	0·17685 (7)	3·1
P(2)	0·25148 (9)	0·06222 (8)	0·01097 (7)	3·1
C(11)	0·3462 (4)	0·0536 (3)	0·1662 (3)	3·3
C(12)	0·3807 (4)	−0·0037 (4)	0·1810 (3)	4·3
C(13)	0·4559 (4)	−0·0122 (4)	0·1730 (4)	5·4
C(14)	0·4984 (4)	0·0353 (5)	0·1502 (4)	6·4
C(15)	0·4665 (4)	0·0925 (4)	0·1337 (4)	6·1
C(16)	0·3907 (4)	0·1006 (4)	0·1421 (3)	4·4
C(21)	0·2008 (3)	−0·0016 (3)	0·2005 (3)	3·1
C(22)	0·2252 (4)	−0·0364 (4)	0·2516 (3)	4·2
C(23)	0·1881 (4)	−0·0909 (4)	0·2670 (3)	4·9
C(24)	0·1269 (5)	−0·1103 (4)	0·2345 (4)	5·3
C(25)	0·1001 (4)	−0·0755 (4)	0·1857 (3)	4·7
C(26)	0·1379 (4)	−0·0207 (3)	0·1691 (3)	3·8
C(31)	0·2463 (4)	0·1171 (3)	0·2490 (3)	3·8
C(32)	0·3005 (4)	0·1590 (5)	0·2636 (4)	6·2
C(33)	0·2957 (5)	0·1956 (5)	0·3162 (5)	8·3
C(34)	0·2368 (6)	0·1879 (5)	0·3557 (4)	8·3
C(35)	0·1822 (5)	0·1473 (5)	0·3414 (4)	7·6
C(36)	0·1866 (4)	0·1116 (4)	0·2881 (4)	5·6
C(41)	0·1960 (4)	0·0404 (4)	−0·0569 (3)	3·9
C(42)	0·1732 (4)	−0·0213 (4)	−0·0665 (4)	5·1
C(43)	0·1236 (5)	−0·0355 (5)	−0·1143 (4)	6·9
C(44)	0·0995 (4)	0·0106 (5)	−0·1526 (4)	6·5
C(45)	0·1237 (4)	0·0704 (5)	−0·1459 (3)	5·8
C(46)	0·1729 (4)	0·0864 (4)	−0·0979 (3)	4·9
C(51)	0·3179 (4)	0·1187 (3)	−0·0204 (3)	3·5
C(52)	0·3595 (4)	0·1053 (4)	−0·0732 (4)	4·9
C(53)	0·4094 (4)	0·1489 (4)	−0·0969 (4)	5·4
C(54)	0·4160 (5)	0·2068 (5)	−0·0700 (4)	6·3
C(55)	0·3736 (4)	0·2224 (4)	−0·0194 (4)	5·5
C(56)	0·3243 (4)	0·1780 (4)	0·0062 (3)	4·3
C(61)	0·3043 (4)	−0·0096 (3)	0·0285 (3)	3·4
C(62)	0·3785 (4)	−0·0168 (4)	0·0156 (3)	4·4
C(63)	0·4139 (4)	−0·0736 (4)	0·0287 (4)	5·7
C(64)	0·3757 (5)	−0·1219 (4)	0·0551 (4)	6·1
C(65)	0·3018 (5)	−0·1155 (4)	0·0690 (4)	5·7
C(66)	0·2661 (4)	−0·0589 (3)	0·0562 (3)	4·1
C(71)	0·1344 (3)	0·1751 (3)	0·1507 (3)	3·2
C(72)	0·1701 (4)	0·2303 (3)	0·1664 (3)	4·1
C(73)	0·1403 (4)	0·2760 (4)	0·2050 (4)	5·3
C(74)	0·0713 (4)	0·2663 (4)	0·2282 (4)	5·8
C(75)	0·0324 (4)	0·2130 (4)	0·2137 (4)	5·5
C(76)	0·0650 (4)	0·1695 (4)	0·1750 (3)	4·2
F(72)	0·2401 (3)	0·2410 (2)	0·1451 (2)	5·5
F(73)	0·1792 (3)	0·3281 (2)	0·2192 (3)	8·2
F(74)	0·0417 (3)	0·3098 (3)	0·2659 (3)	9·6
F(75)	−0·0350 (3)	0·2045 (3)	0·2379 (3)	9·3
F(76)	0·0244 (3)	0·1166 (3)	0·1629 (2)	6·6
C(81)	0·1106 (4)	0·1460 (3)	0·0274 (3)	3·4
C(82)	0·1132 (4)	0·2039 (3)	−0·0019 (3)	4·0
C(83)	0·0597 (4)	0·2227 (4)	−0·0442 (4)	4·8
C(84)	0·0013 (4)	0·1843 (4)	−0·0567 (4)	4·9
C(85)	−0·0048 (4)	0·1274 (4)	−0·0274 (3)	4·2
C(86)	0·0496 (4)	0·1095 (3)	0·0130 (3)	3·6
F(82)	0·1707 (2)	0·2434 (2)	0·0081 (2)	5·6
F(83)	0·0659 (3)	0·2794 (2)	−0·0719 (3)	7·5
F(84)	−0·0503 (3)	0·2025 (3)	−0·0976 (2)	6·6
F(85)	−0·0635 (3)	0·0902 (3)	−0·0391 (2)	6·5
F(86)	0·0425 (3)	0·0520 (2)	0·0396 (2)	5·0

* As defined by Hamilton (1959).

Fourier maps that were based on the position of the Pd atom determined from the Patterson function. The structure was refined by the block-diagonal least-squares procedure using the *HBLS*-V program (Ashida, 1979). 4923 observed reflections [$|F_o| \geq 3\sigma(|F_o|)$] were employed. On the difference Fourier maps after anisotropic refinement, electron densities assigned for all the H atoms were found at essentially the same positions as calculated by stereochemical consideration; these were included in further

Table 2. *Bond distances* (Å) *and bond angles* (°) *for non-*H *atoms with e.s.d.'s in parentheses*

Pd–P(1)	2·343 (2)	Pd–P(2)	2·369 (2)
Pd–C(71)	2·053 (6)	Pd–C(81)	2·059 (6)
P(1)–C(11)	1·820 (7)	P(1)–C(21)	1·832 (6)
P(1)–C(31)	1·844 (7)	P(2)–C(41)	1·838 (7)
P(2)–C(51)	1·824 (7)	P(2)–C(61)	1·835 (7)
C(11)–C(12)	1·401 (9)	C(11)–C(16)	1·383 (9)
C(12)–C(13)	1·380 (11)	C(13)–C(14)	1·358 (12)
C(14)–C(15)	1·389 (12)	C(15)–C(16)	1·390 (11)
C(21)–C(22)	1·400 (9)	C(21)–C(26)	1·384 (9)
C(22)–C(23)	1·377 (10)	C(23)–C(24)	1·374 (11)
C(24)–C(25)	1·375 (10)	C(25)–C(26)	1·394 (10)
C(31)–C(32)	1·357 (11)	C(31)–C(36)	1·374 (10)
C(32)–C(33)	1·381 (13)	C(33)–C(34)	1·373 (14)
C(34)–C(35)	1·344 (14)	C(35)–C(36)	1·381 (12)
C(41)–C(42)	1·386 (10)	C(41)–C(46)	1·382 (11)
C(42)–C(43)	1·400 (12)	C(43)–C(44)	1·354 (13)
C(44)–C(45)	1·350 (13)	C(45)–C(46)	1·408 (12)
C(51)–C(52)	1·397 (10)	C(51)–C(56)	1·389 (9)
C(52)–C(53)	1·388 (11)	C(53)–C(54)	1·362 (12)
C(54)–C(55)	1·376 (12)	C(55)–C(56)	1·408 (10)
C(61)–C(62)	1·377 (9)	C(61)–C(66)	1·388 (9)
C(62)–C(63)	1·391 (11)	C(63)–C(64)	1·361 (12)
C(64)–C(65)	1·374 (12)	C(65)–C(66)	1·390 (11)
C(71)–C(72)	1·379 (9)	C(71)–C(76)	1·364 (9)
C(72)–C(73)	1·386 (10)	C(72)–F(72)	1·363 (8)
C(73)–C(74)	1·359 (11)	C(73)–F(73)	1·344 (9)
C(74)–C(75)	1·365 (11)	C(74)–F(74)	1·343 (10)
C(75)–C(76)	1·377 (11)	C(75)–F(75)	1·336 (10)
C(76)–F(76)	1·366 (8)	C(81)–C(82)	1·383 (9)
C(81)–C(86)	1·380 (9)	C(82)–C(83)	1·389 (10)
C(82)–F(82)	1·349 (8)	C(83)–C(84)	1·358 (10)
C(83)–F(83)	1·346 (9)	C(84)–C(85)	1·368 (10)
C(84)–F(84)	1·341 (9)	C(85)–C(86)	1·369 (9)
C(85)–F(85)	1·342 (8)	C(86)–F(86)	1·354 (8)
P(1)–Pd–P(2)	99·45 (6)	P(1)–Pd–C(71)	88·24 (17)
P(1)–Pd–C(81)	167·36 (17)	P(2)–Pd–C(71)	167·67 (17)
P(2)–Pd–C(81)	89·32 (17)	C(71)–Pd–C(81)	84·7 (3)
Pd–P(1)–C(11)	116·4 (3)	Pd–P(1)–C(21)	108·6 (2)
Pd–P(1)–C(31)	117·1 (3)	C(11)–P(1)–C(21)	108·6 (3)
C(11)–P(1)–C(31)	103·4 (3)	C(21)–P(1)–C(31)	101·5 (3)
Pd–P(2)–C(41)	115·5 (3)	Pd–P(2)–C(51)	107·5 (3)
Pd–P(2)–C(61)	119·2 (2)	C(41)–P(2)–C(51)	103·0 (3)
C(41)–P(2)–C(61)	103·9 (3)	C(51)–P(2)–C(61)	106·2 (3)
P(1)–C(11)–C(12)	125·3 (5)	P(1)–C(11)–C(16)	117·8 (5)
C(12)–C(11)–C(16)	116·9 (6)	C(11)–C(12)–C(13)	121·5 (7)
C(12)–C(13)–C(14)	120·2 (8)	C(13)–C(14)–C(15)	120·4 (8)
C(14)–C(15)–C(16)	118·8 (8)	C(11)–C(16)–C(15)	122·2 (7)
P(1)–C(21)–C(22)	121·0 (5)	P(1)–C(21)–C(26)	119·4 (5)
C(22)–C(21)–C(26)	119·6 (6)	C(21)–C(22)–C(23)	118·7 (7)
C(22)–C(23)–C(24)	121·2 (7)	C(23)–C(24)–C(25)	121·1 (7)
C(24)–C(25)–C(26)	118·3 (7)	C(21)–C(26)–C(25)	121·1 (6)
P(1)–C(31)–C(32)	122·0 (6)	P(1)–C(31)–C(36)	119·4 (5)
C(32)–C(31)–C(36)	118·5 (7)	C(31)–C(32)–C(33)	120·9 (8)
C(32)–C(33)–C(34)	119·7 (9)	C(33)–C(34)–C(35)	119·9 (10)
C(34)–C(35)–C(36)	120·1 (9)	C(31)–C(36)–C(35)	120·8 (8)
P(2)–C(41)–C(42)	121·2 (6)	P(2)–C(41)–C(46)	120·0 (6)
C(42)–C(41)–C(46)	118·7 (7)	C(41)–C(42)–C(43)	120·3 (7)
C(42)–C(43)–C(44)	120·1 (9)	C(43)–C(44)–C(45)	120·5 (9)
C(44)–C(45)–C(46)	120·7 (8)	C(41)–C(46)–C(45)	119·6 (7)
P(2)–C(51)–C(52)	121·6 (5)	P(2)–C(51)–C(56)	119·6 (5)
C(52)–C(51)–C(56)	118·7 (6)	C(51)–C(52)–C(53)	121·0 (7)
C(52)–C(53)–C(54)	120·0 (8)	C(53)–C(54)–C(55)	120·5 (8)
C(54)–C(55)–C(56)	120·3 (7)	C(51)–C(56)–C(55)	119·6 (7)
P(2)–C(61)–C(62)	123·7 (5)	P(2)–C(61)–C(66)	117·1 (5)
C(62)–C(61)–C(66)	119·2 (6)	C(61)–C(62)–C(63)	120·0 (7)
C(62)–C(63)–C(64)	120·3 (8)	C(63)–C(64)–C(65)	120·6 (8)
C(64)–C(65)–C(66)	119·5 (8)	C(61)–C(66)–C(65)	120·3 (7)
Pd–C(71)–C(72)	118·9 (5)	Pd–C(71)–C(76)	127·0 (5)
C(72)–C(71)–C(76)	114·0 (6)	C(71)–C(72)–C(73)	124·1 (6)
C(71)–C(72)–F(72)	119·3 (6)	C(73)–C(72)–F(72)	116·6 (6)
C(72)–C(73)–C(74)	118·2 (7)	C(72)–C(73)–F(73)	120·6 (7)
C(74)–C(73)–F(73)	121·2 (7)	C(73)–C(74)–C(75)	120·7 (8)
C(73)–C(74)–F(74)	119·0 (7)	C(75)–C(74)–F(74)	120·2 (7)
C(74)–C(75)–C(76)	118·3 (7)	C(74)–C(75)–F(75)	119·3 (7)
C(76)–C(75)–F(75)	122·4 (7)	C(71)–C(76)–C(75)	124·7 (7)
C(71)–C(76)–F(76)	119·3 (6)	C(75)–C(76)–F(76)	116·0 (6)
Pd–C(81)–C(82)	125·7 (5)	Pd–C(81)–C(86)	119·4 (5)
C(82)–C(81)–C(86)	114·8 (6)	C(81)–C(82)–C(83)	122·2 (6)
C(81)–C(82)–F(82)	120·2 (6)	C(83)–C(82)–F(82)	117·5 (6)
C(82)–C(83)–C(84)	119·9 (7)	C(82)–C(83)–F(83)	119·4 (6)
C(84)–C(83)–F(83)	120·7 (7)	C(83)–C(84)–C(85)	120·0 (7)
C(83)–C(84)–F(84)	119·8 (7)	C(85)–C(84)–F(84)	120·2 (7)
C(84)–C(85)–C(86)	118·8 (7)	C(84)–C(85)–F(85)	119·6 (6)
C(86)–C(85)–F(85)	121·6 (6)	C(81)–C(86)–C(85)	124·2 (6)
C(81)–C(86)–F(86)	118·9 (6)	C(85)–C(86)–F(86)	116·9 (6)

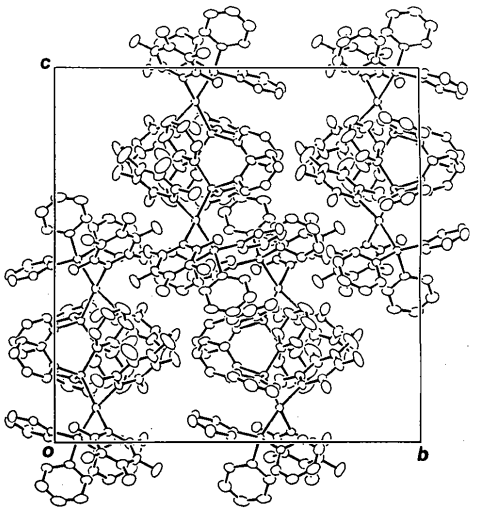

Fig. 2. *ORTEP* drawing (Johnson, 1976) of the crystal packing diagram as viewed along the *a* axis. Non-H atoms are represented by thermal ellipsoids with 30% probability levels; H atoms are omitted for clarity.

refinements. The function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w = [\sigma(F_o)^2 + a|F_o| + b|F_o|^2]^{-1}$ with $a = 0{\cdot}0064$ and $b = 0{\cdot}0001$ in the final refinement cycle. The number of observations per refined parameter is $4923/671 = 7{\cdot}34$; $S = 1{\cdot}12$. The final R and wR values are 0·053 and 0·056, respectively. $(\Delta/\sigma)_{max}$ was 0·54 for non-H atoms in the final refinement cycle. The peaks in the final $\Delta\rho$ map were between 0·3 and $-0{\cdot}3$ e Å^{-3} except in the region around the Pd atom, where peaks between 0·6 and $-0{\cdot}4$ e Å^{-3} were observed. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The final atomic parameters are listed in Table 1.* The molecular structure with atomic numbering system and the crystal packing diagram are depicted in Figs. 1 and 2, respectively. Bond distances and angles are presented in Table 2.

*Lists of anisotropic temperature factors for non-H atoms, atomic parameters for H atoms, and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44734 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

All the computations were performed on an ACOS 850 computer at the Crystallographic Research Center, Institute for Protein Research, Osaka University.

Related literature. The preparation of the title compound has been reported (Usón, Forniés, Espinet, Martínez & Tomás, 1981). The Pd—P bond lengths and the P—Pd—P bond angle in the present complex fall in the range of known values (2·27–2·38 Å, 98·1–108·1°) found in Pd^{II} complexes containing two *cis*-triphenylphosphine ligands (Ahmed, Itoh, Matsuda, Ueda, Ishii & Ibers, 1977; Godleski, Gundlach, Ho, Keinan & Frolow, 1984; Jones, Sheldrick, Usón, Forniés & Usón, 1983; Mealli, Midollini, Moneti, Sacconi, Silvestre & Albright, 1982; Miki, Kai, Yasuoka & Kasai, 1981; Zenitani, Tokunan, Kai, Yasuoka & Kasai, 1978). The structures of Pd^{II} complexes containing the Pd—C_6F_5 bond have been reported by Usón, Forniés, Navarro, Usón, Garcia & Welch (1984) [Pd—C = 2·057 Å] and by Usón, Laguna, Forniés, Valenzuela, Jones & Sheldrick (1984) [Pd—C = 2·106 Å].

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