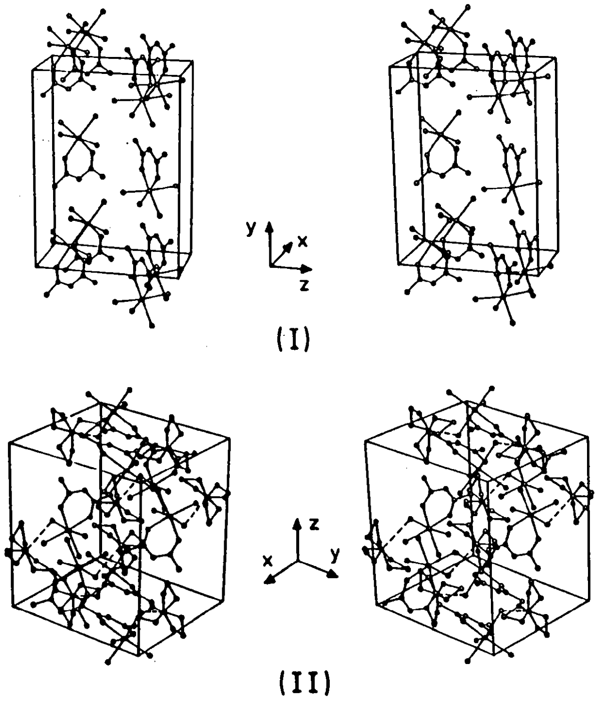

(I)

(II)

Fig. 2. Stereo plots of the crystal structures. Atoms with arbitrary size (H atoms omitted), hydrogen bonds with dotted lines.

& Schmidtke, 1986). Crystal structure analyses of related compounds: $Sb(acac)MeCl_3$, $Sb(acac)Me_2Br_2$, $Sb(acac)Ph_2Cl_2$ (Kanehisa, Onuma, Uda, Hirabayashi, Kai, Yasuoka & Kasai, 1978); $Sn(acac)_2Me_2$, $Sn(acac)_2Cl_2$ (Miller & Schlemper, 1973, 1978).

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(η-Cyclopentadienyl)[methoxy(methyl)carbene]bis(triphenylphosphine)ruthenium(II) Hexafluorophosphate

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Abstract. $C_{44}H_{41}F_6OP_3Ru$, $M_r = 893{\cdot}8$, orthorhombic, $P2_12_12_1$, $a = 11{\cdot}934\,(4)$, $b = 14{\cdot}858\,(4)$, $c = 22{\cdot}287\,(4)$ Å, $V = 3952\,(2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}502$ Mg m^{-3}, Mo $K\bar{\alpha}$ radiation, $\lambda = 0{\cdot}7107$ Å, $\mu = 0{\cdot}531$ mm^{-1}, $F(000) = 1824$, $T = 293\,(2)$ K, $R = 0{\cdot}046$ for 3219 observed reflections. The crystal structure of the title compound is comprised of discrete $[Ru\{C(OCH_3)CH_3\}\{P(C_6H_5)_3\}_2(\eta\text{-}C_5H_5)]^+$ cations and $[PF_6]^-$ anions. In the cation the Ru atom exists in a distorted octahedral environment with one face being occupied by the η-C_5H_5 ring and the other by the two P atoms [Ru—P 2·333 (2), 2·336 (2) Å; P—Ru—P 101·3 (1)°] and C(6), at 1·931 (9) Å, of the $C(OCH_3)CH_3$ group [P—Ru—C 86·6 (2), 89·8 (2)°].

Experimental. The complex was prepared in 62% yield from $[Ru(NCCH_3)\{P(C_6H_5)_3\}_2(\eta\text{-}C_5H_5)][PF_6]$ and $HC_2Si(CH_3)_3$ in CH_3OH; crystals were obtained from $CH_3OH/CDCl_3$. Found: C, 58·9; H, 4·6; $C_{44}H_{41}F_6OP_3Ru$ requires C, 58·9; H, 4·6%. IR (Nujol): ν(CO) 1270*s*, ν(PF) 840*vs*(*br*). ^{1}H NMR: $\delta(CDCl_3$; p.p.m.) 3·02*s* (CH_3), 3·28*s* (OCH_3), 4·75*s* (C_5H_5), 7·0–7·4*m* (C_6H_5). ^{13}C NMR: $\delta(CDCl_3$; p.p.m.) 46·4*s* (CH_3), 60·7*s* (OCH_3), 91·4*s* (C_5H_5), 128·2–136·1*m* (C_6H_5), 309·2*t*, *J*(CP) 0·2Hz (Ru*C*). FAB MS: M$^+$ at *m*/*z* 749. Enraf–Nonius CAD-4F diffractometer controlled by a PDP8/A computer, graphite-monochromated Mo $K\bar{\alpha}$ radiation; ω:2θ scan technique. Cell parameters on crystal 0·50 × 0·18 × 0·50 mm from least-squares

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Table 1. *Fractional atomic coordinates and B_{eq} values* (Å^2)

$$B_{\mathrm{eq}} = 8\pi^2(U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	B_{eq}
Ru	−0·19498 (5)	0·00252 (4)	−0·18004 (3)	2·46
P(1)	−0·3115 (2)	−0·0746 (1)	−0·1135 (1)	2·42
P(2)	−0·0654 (2)	−0·1093 (1)	−0·2047 (1)	2·67
P(3)	−0·6045 (3)	−0·1339 (2)	0·3909 (1)	6·06
F(1)	−0·5514 (9)	−0·2142 (6)	−0·3544 (4)	10·65
F(2)	−0·5471 (11)	−0·0685 (6)	−0·3487 (7)	16·81
F(3)	−0·6581 (8)	−0·0565 (5)	−0·4290 (4)	10·00
F(4)	−0·4981 (9)	−0·1341 (8)	−0·4298 (6)	16·28
F(5)	−0·6482 (12)	−0·1975 (6)	−0·4364 (6)	18·13
F(6)	−0·7068 (9)	−0·1392 (10)	−0·3534 (6)	18·73
O(1)	−0·0396 (5)	−0·0255 (4)	−0·0851 (3)	3·50
C(1)	−0·2806 (7)	0·0201 (6)	−0·2713 (4)	4·06
C(2)	−0·3399 (8)	0·0680 (7)	−0·2270 (5)	4·77
C(3)	−0·2723 (8)	0·1369 (6)	−0·2026 (4)	4·59
C(4)	−0·1688 (8)	0·1304 (6)	−0·2307 (5)	4·92
C(5)	−0·1703 (8)	0·0603 (6)	−0·2724 (4)	4·39
C(6)	−0·1059 (7)	0·0343 (5)	−0·1109 (4)	3·22
C(7)	0·0310 (7)	−0·0043 (7)	−0·0344 (4)	5·32
C(8)	−0·1045 (9)	0·1252 (6)	−0·0819 (6)	6·05
C(9)	−0·3667 (4)	−0·1840 (3)	−0·1388 (2)	2·66
C(10)	−0·4026 (4)	−0·1896 (3)	−0·1983 (2)	4·10
C(11)	−0·4383 (4)	−0·2718 (3)	−0·2216 (2)	5·13
C(12)	−0·4380 (4)	−0·3485 (3)	−0·1856 (2)	4·45
C(13)	−0·4021 (4)	−0·3430 (3)	−0·1261 (2)	4·32
C(14)	−0·3664 (4)	−0·2607 (3)	−0·1028 (2)	3·31
C(15)	−0·2583 (4)	−0·0938 (3)	−0·0371 (2)	2·57
C(16)	−0·1902 (4)	−0·1678 (3)	−0·0245 (2)	3·63
C(17)	−0·1485 (4)	−0·1804 (3)	0·0333 (2)	5·54
C(18)	−0·1748 (4)	−0·1191 (3)	0·0786 (2)	4·82
C(19)	−0·2428 (4)	−0·0451 (3)	0·0660 (2)	4·56
C(20)	−0·2845 (4)	−0·0325 (3)	0·0082 (2)	3·33
C(21)	−0·4444 (3)	−0·0187 (3)	−0·0930 (2)	2·72
C(22)	−0·4477 (3)	0·0748 (3)	−0·0880 (2)	3·91
C(23)	−0·5423 (3)	0·1169 (3)	−0·0641 (2)	4·72
C(24)	−0·6334 (3)	0·0654 (3)	−0·0452 (2)	5·35
C(25)	−0·6300 (3)	−0·0281 (3)	−0·0502 (2)	4·72
C(26)	−0·5355 (3)	−0·0702 (3)	−0·0740 (2)	4·19
C(27)	−0·0790 (4)	−0·1445 (3)	−0·2852 (2)	2·95
C(28)	−0·1813 (4)	−0·1790 (3)	−0·3047 (2)	3·91
C(29)	−0·1968 (4)	−0·2000 (3)	−0·3652 (2)	5·39
C(30)	−0·1099 (4)	−0·1864 (3)	−0·4061 (2)	5·91
C(31)	−0·0076 (4)	−0·1518 (3)	−0·3865 (2)	5·10
C(32)	0·0079 (4)	−0·1309 (3)	−0·3261 (2)	3·95
C(33)	−0·0543 (4)	−0·2184 (3)	−0·1641 (2)	2·56
C(34)	−0·1209 (4)	−0·2914 (3)	−0·1807 (2)	3·96
C(35)	−0·1136 (4)	−0·3723 (3)	−0·1491 (2)	4·66
C(36)	−0·0396 (4)	−0·3801 (3)	−0·1009 (2)	4·90
C(37)	0·0270 (4)	−0·3071 (3)	−0·0843 (2)	5·07
C(38)	0·0197 (4)	−0·2263 (3)	−0·1159 (2)	4·09
C(39)	0·0807 (4)	−0·0717 (3)	−0·2035 (2)	3·14
C(40)	0·1666 (4)	−0·1350 (3)	−0·2086 (2)	4·41
C(41)	0·2782 (4)	−0·1069 (3)	−0·2088 (2)	5·62
C(42)	0·3038 (4)	−0·0156 (3)	−0·2038 (2)	5·55
C(43)	0·2179 (4)	0·0476 (3)	−0·1987 (2)	4·79
C(44)	0·1064 (4)	0·0196 (3)	−0·1985 (2)	3·78

Table 2. *Interatomic distances* (Å) *and bond angles* (°)

Ru—P(1)	2·333 (2)	Ru—P(2)	2·336 (2)
Ru—C(1)	2·292 (8)	Ru—C(2)	2·244 (8)
Ru—C(3)	2·257 (8)	Ru—C(4)	2·232 (8)
Ru—C(5)	2·250 (8)	Ru—C(6)	1·931 (9)
C(6)—O(1)	1·321 (9)	C(6)—C(8)	1·50 (1)
O(1)—C(7)	1·44 (1)		
P(1)—Ru—P(2)	101·3 (1)	P(1)—Ru—C(6)	86·6 (2)
P(2)—Ru—C(6)	89·8 (2)	Ru—C(6)—O(1)	120·9 (6)
Ru—C(6)—C(8)	124·8 (7)	O(1)—C(6)—C(8)	114·3 (8)
C(6)—O(1)—C(7)	122·9 (7)		

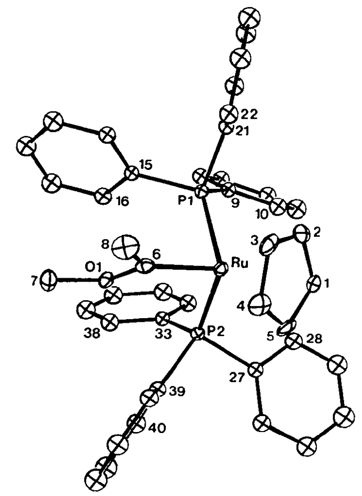

Fig. 1. Molecular structure and numbering scheme for the $[\mathrm{Ru\{C(OCH_3)CH_3\}\{P(C_6H_5)_3\}_2(\eta\text{-}C_5H_5)}]^+$ cation showing 15% probability ellipsoids. Atoms not otherwise indicated are carbons (Johnson, 1971).

procedure (de Boer & Duisenberg, 1984) on 25 reflections ($2 \leq \theta \leq 8°$). Total of 4419 reflections ($1{\cdot}5 \leq \theta \leq 25°$) measured in the range $-14 \leq h \leq 0$, $-17 \leq k \leq 0$, $-23 \leq l \leq 3$; some high-angle Friedel pairs were also included. No significant variation in the net intensities of three reference reflections ($\bar{2}\bar{2}2$, $\bar{1}2\bar{1}$, $0\bar{2}1$) measured every 3600 s. 4300 unique reflections (R_{int} 0·021), 3219 satisfied $I \geq 2{\cdot}5\sigma(I)$. Analytical absorption correction applied (Sheldrick, 1976); max./min. transmission factors 0·860 and 0·794. Structure solved by Patterson, full-matrix least-squares refinement of 247 parameters based on F (Sheldrick, 1976). Phenyl rings refined as hexagonal rigid groups, anisotropic thermal parameters for remaining non-H atoms and H atoms included at their calculated positions. At convergence $R = 0{\cdot}046$ (preferred chirality), $wR = 0{\cdot}049$, $w = 4{\cdot}4/[\sigma^2(F) + 0{\cdot}0006F^2]$, $S = 4{\cdot}0$, $(\Delta/\sigma)_{\max} \leq 0{\cdot}19$ (for $\mathrm{PF_6}$ anion), $(\Delta\rho)_{\max} = 0{\cdot}79$, $(\Delta\rho)_{\min} = -0{\cdot}50$ e Å^{-3}; no extinction correction. Scattering factors for C, H, F, O and P given in *SHELX*76 (Sheldrick, 1976) and those for neutral Ru corrected for f' and f'' (Hamilton & Ibers, 1974). All calculations on VAX11/785 computer system. Atomic parameters given in Table 1, selected bond distances and angles in Table 2;* the numbering scheme used is shown in Fig. 1.

Related literature. The Ru—C(6) bond distance of 1·931 (9) Å is similar to 1·959 (6) Å found in the isomorphous $[\mathrm{Ru\{C(OCH_3)C_2H_5\}\{P(C_6H_5)_3\}_2(\eta\text{-}C_5H_5)}][\mathrm{PF_6}]$ compound (Bruce, Humphrey, Snow & Tiekink, 1986); other comparable geometric parameters are also equivalent in these structures.

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* Lists of structure factors, thermal parameters, H-atom parameters and interatomic distances and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44826 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

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Abstract. $[Pd\{(C_6H_5)_3P\}_2(C_6F_5)_2]$, $M_r = 965{\cdot}1$, 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 $K\alpha$) $= 0{\cdot}71069$ 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_6F_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_2Cl_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

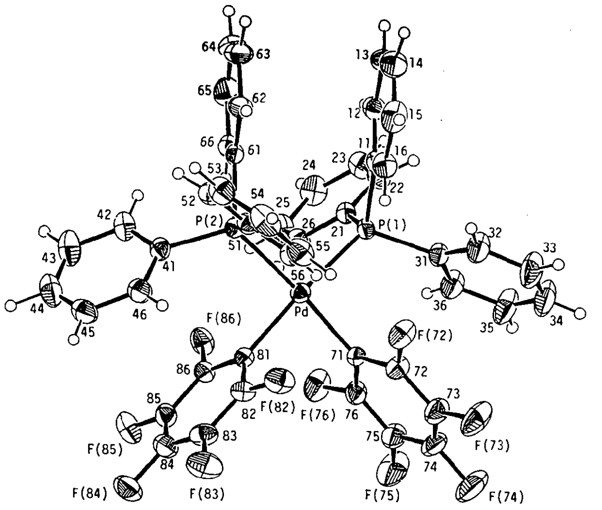

Fig. 1. *ORTEP* drawing (Johnson, 1976) of the molecular structure together with atomic numbering system. For carbon atoms only atomic numbers are shown for clarity. Non-H atoms are represented by thermal ellipsoids with 30% probability levels; H atoms are drawn by a sphere with $B = 1{\cdot}0$ Å^2.

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