cis,cis,cis-(Acetato-O,O′)bis[1,2-bis(diphenylphosphino)ethane-P,P′]ruthenium(II) hexafluorophosphate dimethanol solvate

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cis,cis,cis-(Acetato-O,O')bis[1,2-bis-(diphenylphosphino)ethane-P,P']-ruthenium(II) hexafluorophosphate dimethanol solvate

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In the cation of the title complex, cis,cis,cis-[Ru(η^2-O_2CMe)(dppe)$_2$]PF$_6$·2MeOH [dppe is 1,2-bis(diphenylphosphino)ethane, $C_{26}H_{24}P_2$], the Ru atom is in a pseudo-octahedral coordination environment with two chelating dppe ligands and one chelating acetate ligand. Intra-phosphine and intra-acetate bond lengths and angles are unexceptional. Deviations from idealized octahedral coordination angles at ruthenium [O—Ru—O 59.43 (8)° and P—Ru—P 103.19 (2)°] presumably derive from constraints imposed by the chelate rings. The Ru—P distances for the mutually trans P-donor atoms [2.3785 (6) Å] are significantly longer than those for the Ru—P linkages trans to the acetate ligand [2.3074 (6) Å]. The Ru1, C1 and C2 atoms lie on a twofold axis, and atom P3 of the anion lies on an inversion centre.

Comment

For the most part, the bond distances and angles of the title complex, (I), are unremarkable. Deviations from idealized octahedral coordination angles at ruthenium [O1—Ru1—O1^i 59.43 (8)° and P1—Ru1—P2 103.19 (2)°; symmetry code: (i) $2 - x$, y, $\frac{3}{2} - z$] presumably derive from constraints imposed

(I)
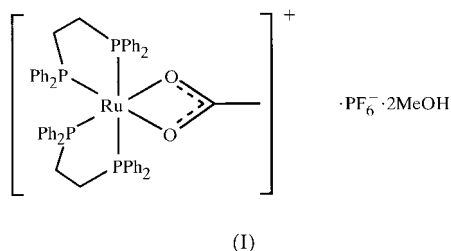
by the chelate rings, as similar angles have been observed in ruthenium complexes with chelating acetate (Boyar et al., 1986) or dppe (Bruce et al., 1986) ligands in earlier work. The Ru—P distances for the mutually trans P-donor

[2.3785 (6) Å] are significantly longer than those for the Ru—P linkages trans to acetate [2.3074 (6) Å], a result noted previously in the related complex cis,cis,cis-[Ru(η^2-O_2CMe)(dppm)$_2$]BPh$_4$ [dppm is bis(diphenylphosphino)methane] and ascribed to the varying trans influence of the phosphine and acetate ligands (Boyar et al., 1986).

Experimental

The title complex was prepared by refluxing a solution of cis-[RuCl$_2$(dppe)$_2$], sodium acetate (20 molar equivalents), and ammonium hexafluorophosphate (1.5 molar equivalents) in dichloromethane for 3 h. The solvent was then removed under reduced pressure and the title complex was purified by being passed through an alumina plug with acetone. It was then recrystallized from dichloromethane/methanol. The following characterization data were collected: MS: 957 ([$M^+ - PF_6$], 100), 898 ([$M^+ - PF_6 - CH_3COO$], 75); ^{1}H NMR (δ, 300 MHz, CDCl$_3$): 6.88–7.79 (m, 40H, Ph), 2.96 (m, 2H, CH$_2$), 2.24 (m, 2H, CH$_2$), 1.98 (m, 2H, CH$_2$), 1.47 (m, 2H, CH$_2$), 0.49 (s, 3H, CH$_3$); ^{31}P NMR (δ, 121 MHz, CDCl$_3$): 60.0 (t, J_{PP} = 18 Hz, PCH$_2$), 59.2 (t, J_{PP} = 18 Hz, PCH$_2$), −144.3 (sept, J_{PF} = 713 Hz, PF$_6$); analysis calculated for $C_{54}H_{51}F_6O_2P_5Ru$·2CH$_4$O: C 57.69, H 5.10%; found: C 57.38, H 4.85%.

Crystal data

[Ru(C$_2$H$_3$O$_2$)(C$_{26}$H$_{24}$P$_2$)$_2$]PF$_6$·2CH$_4$O	D_x = 1.457 Mg m^{-3}
M_r = 1166.01	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 41382 reflections
a = 13.4832 (2) Å	θ = 2.910–27.485°
b = 19.6340 (2) Å	μ = 0.512 mm^{-1}
c = 20.3112 (3) Å	T = 273.2 K
β = 98.6340 (8)°	Block, orange
V = 5316.0 (1) Å^3	0.30 × 0.20 × 0.15 mm
Z = 4	

Data collection

KappaCCD diffractometer	6127 independent reflections
ω scans	4464 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan [SORTAV (Blessing, 1995, 1997) implemented in maXus (Mackay et al., 1999)]	R_{int} = 0.068
	θ_{max} = 27.51°
	$h = -17 \rightarrow 17$
	$k = -25 \rightarrow 25$
T_{min} = 0.847, T_{max} = 0.926	$l = -25 \rightarrow 26$
53 848 measured reflections	

Refinement

Refinement on F	H atoms treated by a mixture of independent and constrained refinement
R = 0.035	
wR = 0.039	
S = 0.949	$w = 1/[\sigma^2(F_o) + 0.00040|F_o|^2]$
4464 reflections	$(\Delta/\sigma)_{max}$ = 0.0217
328 parameters	$\Delta\rho_{max}$ = 0.49 e Å^{-3}
	$\Delta\rho_{min}$ = −0.66 e Å^{-3}

The H atoms of the ruthenium cation were placed in calculated positions. The solvent methyl H atoms were placed at calculated positions based on peaks from the Fourier difference map, while the alcoholic proton was located from the difference map and refined with a restraint on the O—H distance.

Data collection: COLLECT (Nonius, 1999); cell refinement: DENZO–SMN (Otwinowski & Minor, 1997); data reduction: DENZO–SMN; program(s) used to solve structure: SIR92 (Altomare et al., 1994); program(s) used to refine structure: TEXSAN (Molecular Structures Corporation, 1997); software used to prepare material for publication: TEXSAN.

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