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Key indicators

Single-crystal X-ray study
 T = 200 K
 Mean $\sigma(\text{C}-\text{C}) = 0.007 \text{ \AA}$
 H-atom completeness 84%
 Disorder in solvent or counterion
 R factor = 0.032
 wR factor = 0.036
 Data-to-parameter ratio = 13.4

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

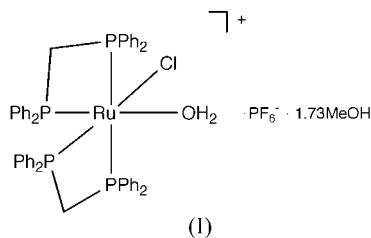
***cis,cis,cis*-Aquabis[bis(diphenylphosphino)methane- κ^2P,P']chlororuthenium(II) hexafluorophosphate methanol 1.73-solvate**

The title compound, $[\text{RuCl}(\text{C}_{25}\text{H}_{22}\text{P}_2)_2(\text{H}_2\text{O})]\text{PF}_6 \cdot 1.73\text{CH}_4\text{O}$, contains a pseudo-octahedral ruthenium complex cation with two bidentate diphosphine ligands and *cis*-disposed chloro and aqua ligands, together with a disordered hexafluorophosphate anion and disordered methanol solvent molecules. Identification as an aquaruthenium(II) complex rather than a hydroxoruthenium(III) complex was confirmed by cyclic voltammetry.

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Comment

For the most part, the bond distances and angles of the title complex, (I), are unremarkable and similar to those of related complexes (Hartwig *et al.*, 1991). Deviations from octahedral geometry at the Ru atom result from the presence of two chelating bis(diphenylphosphino)methane ligands [P1—Ru1—P2 = 71.08 (3)° and P3—Ru1—P4 = 71.33 (3)°]. The Ru—P distances for the mutually *trans* P-donor atoms [Ru1—P1 = 2.345 (1) Å and Ru1—P3 = 2.3839 (9) Å] are significantly longer than those *trans* to the chloro and aqua ligands [Ru1—P2 = 2.3028 (8) Å and Ru1—P4 = 2.312 (1) Å].



Experimental

A solution of *cis*- $[\text{RuCl}_2(\text{dppm})_2]$ (50 mg, 0.053 mmol), sodium hexafluorophosphate (15 mg, 0.089 mmol), water (2 ml) and triethylamine (2 ml) in dichloromethane was stirred for 16 h. The solvent was removed under reduced pressure and the title complex was purified by passing a dichloromethane extract of the residue through an alumina plug with dichloromethane. Compound (I) was then recrystallized from dichloromethane/methanol, affording green crystals (yield 42.5 mg, 75%). Analysis calculated for $\text{C}_{50}\text{H}_{46}\text{ClF}_6\text{O}-\text{P}_5\text{Ru} \cdot 1.73\text{CH}_4\text{O}$: C 55.34, H 4.65%; found: C 55.39, H 4.28%. IR (CH_2Cl_2): ν_{PF_6} 846 cm^{-1} . ^1H NMR (300 MHz, CDCl_3): δ 6.35–8.20 (*m*, 40, Ph), 5.30 (*m*, 4, CH_2). UV–vis: $\lambda(\text{THF})$ 346 nm, ϵ 600 $\text{M}^{-1} \text{cm}^{-1}$. E-chem: $\text{Ru}^{\text{II/III}} = 0.51 \text{ V}$, $\text{Ru}^{\text{III/IV}} = 1.29 \text{ V}$.

Crystal data

$[\text{RuCl}(\text{C}_{25}\text{H}_{22}\text{P}_2)_2(\text{H}_2\text{O})]\text{PF}_6 \cdot 1.73\text{CH}_4\text{O}$	$D_x = 1.474 \text{ Mg m}^{-3}$
$M_r = 1123.67$	Mo $K\alpha$ radiation
Monoclinic, <i>Cc</i>	Cell parameters from 50 753 reflections
$a = 20.4427 (2) \text{ \AA}$	$\theta = 3\text{--}27^\circ$
$b = 12.7593 (2) \text{ \AA}$	$\mu = 0.58 \text{ mm}^{-1}$
$c = 21.0262 (2) \text{ \AA}$	$T = 200 \text{ K}$
$\beta = 112.6186 (7)^\circ$	Block, green
$V = 5062.53 (11) \text{ \AA}^3$	$0.30 \times 0.26 \times 0.17 \text{ mm}$
$Z = 4$	

Data collection

Nonius KappaCCD diffractometer
 φ and ω scans
 Absorption correction: by integration [Coppens (1970); implemented in *maXus* (Mackay *et al.*, 2000)]
 $T_{\min} = 0.883$, $T_{\max} = 0.957$
 50 753 measured reflections

Refinement

Refinement on F
 $R = 0.032$
 $wR = 0.036$
 $S = 1.05$
 8523 reflections
 637 parameters
 H-atom parameters constrained
 Chebychev polynomial with five parameters: 0.771, 0.341, 0.66,

11 446 independent reflections
 8523 reflections with $I > 3\sigma(I)$
 $R_{\text{int}} = 0.05$
 $\theta_{\text{max}} = 27.5^\circ$
 $h = -26 \rightarrow 26$
 $k = -16 \rightarrow 16$
 $l = -27 \rightarrow 27$

0.079 and 0.197 (Carruthers & Watkin, 1979)
 $(\Delta/\sigma)_{\text{max}} = 0.019$
 $\Delta\rho_{\text{max}} = 0.69 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -1.37 \text{ e } \text{\AA}^{-3}$
 Absolute structure: Flack (1983),
 5819 Friedel pairs
 Flack parameter = -0.02 (2)

Table 1

Selected geometric parameters (\AA , $^\circ$).

Ru1—Cl1	2.4656 (8)	P2—C13	1.818 (3)
Ru1—P1	2.345 (1)	P2—C19	1.836 (4)
Ru1—P2	2.3028 (8)	P2—C49	1.857 (3)
Ru1—P3	2.3839 (9)	P3—C25	1.839 (3)
Ru1—P4	2.312 (1)	P3—C31	1.820 (3)
Ru1—O1	2.196 (3)	P3—C50	1.830 (4)
P1—C1	1.816 (4)	P4—C37	1.827 (4)
P1—C7	1.827 (4)	P4—C43	1.824 (4)
P1—C49	1.830 (3)	P4—C50	1.836 (3)
Cl1—Ru1—P1	92.74 (3)	P3—Ru1—P4	71.33 (3)
Cl1—Ru1—P2	163.69 (3)	Cl1—Ru1—O1	81.41 (9)
P1—Ru1—P2	71.08 (3)	P1—Ru1—O1	88.92 (9)
Cl1—Ru1—P3	93.70 (3)	P2—Ru1—O1	95.83 (9)
P1—Ru1—P3	172.00 (4)	P3—Ru1—O1	96.74 (9)
P2—Ru1—P3	102.60 (3)	P4—Ru1—O1	166.73 (9)
Cl1—Ru1—P4	93.34 (4)	P1—C49—P2	94.24 (15)
P1—Ru1—P4	103.56 (4)	P3—C50—P4	96.68 (18)
P2—Ru1—P4	92.54 (3)		

The crystallographic asymmetric unit consists of one $[\text{RuCl}(\text{OH}_2)(\text{PPh}_2\text{CH}_2\text{PPh}_2)_2]^+$ cation, one PF_6^- anion and methanol molecules of solvation at two general locations. One methanol molecule (atoms O3 and C52) appears to have an occupancy of less than 1.0. When it is present, the other methanol is sited at O2 and C51, and the hexafluorophosphate F atoms are at F1—F6; when it is absent, the other methanol is at O21 and C511, and the hexafluorophosphate F atoms occupy sites F1, F21, F31, F4, F51 and F61, corresponding to a rotation about the F1—P5—F4 axis. The relative occupancies of these two alternatives were refined, the final values being 0.730 (5):0.270 (5). The O2—C51 and O21—C511 distances were restrained to be equal. The largest peaks in the final difference electron density map are located near the Ru atom. The space group is non-centrosymmetric but contains glide planes, giving a racemic structure. H atoms attached to C atoms of the cation were included at idealized positions and allowed to ride on the atoms to which they are bonded, with C—H = 1.0 \AA and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. H atoms of the aqua ligand and of the methanol molecules were not located.

Data collection: *COLLECT* (Nonius, 1997); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data

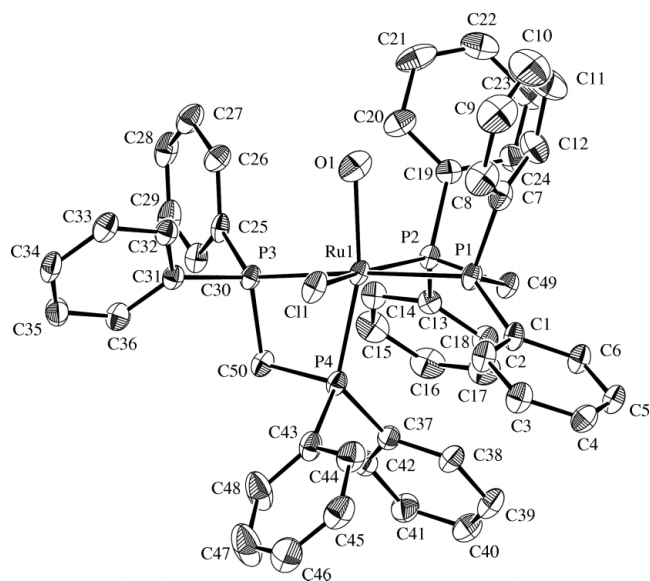


Figure 1

A view of the cation of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted.

reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Watkin *et al.*, 2001); molecular graphics: *ORTEPII* (Johnson, 1976) in *TEXSAN* (MSC, 1992–1997); software used to prepare material for publication: *CRYSTALS*.

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