

**cis-Aquabis[bis(diphenylphosphino)ethane- $\kappa_2P,P'$ ]-chlororuthenium(II) hexafluorophosphate dichloromethane sesquisolvate hemihydrate**Luca Russo,<sup>a</sup> João Figueira,<sup>b</sup> João Rodrigues<sup>b</sup> and Kari Rissanen<sup>a\*</sup><sup>a</sup>Nanoscience Centre, Department of Chemistry, University of Jyväskylä, PO Box 35, 40014 Jyväskylä, Finland, and <sup>b</sup>Centro de Química da Madeira, LQCM, Universidade da Madeira, 9000-390 Funchal, Portugal

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**Key indicators**Single-crystal X-ray study  
 $T = 173$  K  
Mean  $\sigma(C-C) = 0.011$  Å  
H-atom completeness 99%  
Disorder in solvent or counterion  
 $R$  factor = 0.067  
 $wR$  factor = 0.140  
Data-to-parameter ratio = 13.6For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

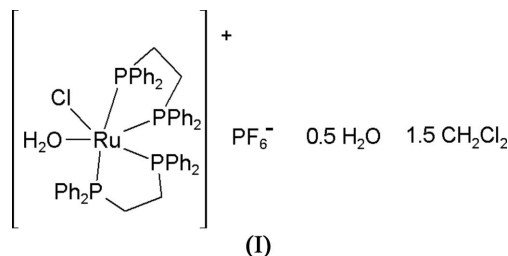
In the title compound,  $[RuCl(C_{26}H_{24}P)_2(H_2O)]PF_6 \cdot 1.5CH_2Cl_2 \cdot 0.5H_2O$ , the complex  $Ru^{II}$  cation is in a slightly distorted octahedral environment, chelated by two bis(diphenylphosphino)ethane ligands, with a water molecule and a chloride anion in a mutually *cis* geometry completing the coordination.

Received 21 February 2006

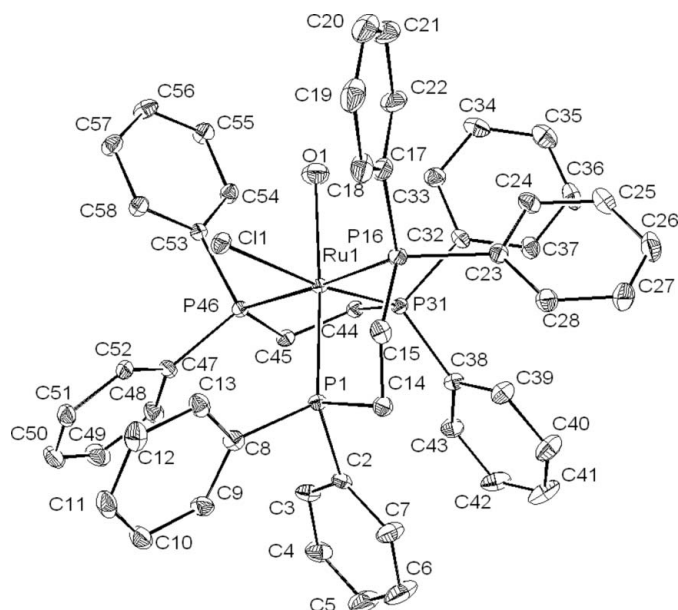
Accepted 25 February 2006

**Comment**

A number of examples of structures in which  $Ru^{II}$  is coordinated by two bis(diphenylphosphino)ethane ligands and chloride anions can be found in the literature. In two cases the metal centre is five-coordinate, with only one chloride and with  $BF_4^-$  (Polam & Porter, 1993) or  $PF_6^-$  (Chin *et al.* 1994) acting as counter-ion. In four more cases the metal is six-coordinate, with an additional *trans* chloride ion completing the coordination environment; the unsolvated compound has been reported (Polam & Porter, 1993), along with the structures of the dichloromethane (Lobana *et al.*, 1990), chloroform (Fronczek *et al.*, 2001) and tetrahydrofuran (THF; Chang *et al.*, 1998) solvates. In several other cases a different ligand coordinates *trans* to the chloride anion, to complete the octahedral coordination. The title compound, (I), is the first example where the sixth ligand, a water molecule, coordinates *cis* to the chloride; the same coordination environment is, however, observed in a known structure (Willis *et al.*, 2004), where the metal is chelated by bis(diphenylphosphino)methane ligands rather than bis(diphenylphosphino)ethane. Bond lengths and angles within the distorted octahedral coordination environment of the metal are reported in Table 1. Along with the counter-ion  $PF_6^-$ , the asymmetric unit also contains 1.5 molecules of dichloromethane and 0.5 molecule of uncoordinated water.

**Experimental**

The title compound, (I), was obtained as a by-product of the reaction between a solution of *cis*- $[Ru(P_2C_{26}H_{24})_2Cl_2]$  (0.280 g, 0.29 mmol),  $NH_4PF_6$  (pre-dried under high vacuum, 0.082 g, 0.50 mmol) in pre-



**Figure 1**  
Structure of the complex cation showing the atom numbering scheme and with H atoms omitted. Displacement ellipsoids are drawn at the 30% probability level.

dried THF (20 ml) and a solution of 1,4-dioxy-2,5-diethynylbenzene (0.025 g, 0.11 mmol), dissolved in pre-dried THF (2 ml) with triethylamine (0.5 ml, 3.6 mmol). After 24 h of stirring, the yellow solution was filtered and the solvent was removed under reduced pressure, yielding a dark-yellow oil. After washing with dry *n*-hexane and drying under vacuum, the yellow solid was recrystallized from dichloromethane/*n*-hexane (10:3), giving bright-yellow crystals.

**Crystal data**

[RuCl(C<sub>26</sub>H<sub>24</sub>P)<sub>2</sub>(H<sub>2</sub>O)]PF<sub>6</sub>·  
1.5CH<sub>2</sub>Cl<sub>2</sub>·0.5H<sub>2</sub>O  
M<sub>r</sub> = 1233.70  
Monoclinic, C2/c  
a = 37.707 (12) Å  
b = 14.552 (3) Å  
c = 25.583 (5) Å  
β = 129.777 (19)°  
V = 10789 (5) Å<sup>3</sup>  
Z = 8  
D<sub>x</sub> = 1.519 Mg m<sup>-3</sup>  
Mo Kα radiation  
Cell parameters from 8488 reflections  
θ = 2.5–27.5°  
μ = 0.70 mm<sup>-1</sup>  
T = 173 (2) K  
Plate, yellow  
0.40 × 0.15 × 0.04 mm

**Data collection**

Nonius KappaCCD diffractometer  
φ and ω scans  
Absorption correction: multi-scan  
(SADABS; Sheldrick, 2003)  
T<sub>min</sub> = 0.768, T<sub>max</sub> = 0.973  
66705 measured reflections  
9402 independent reflections  
6671 reflections with I > 2σ(I)  
R<sub>int</sub> = 0.129  
θ<sub>max</sub> = 25.0°  
h = -44 → 44  
k = -17 → 16  
l = -30 → 30

**Refinement**

Refinement on F<sup>2</sup>  
R[F<sup>2</sup> > 2σ(F<sup>2</sup>)] = 0.067  
wR(F<sup>2</sup>) = 0.140  
S = 1.10  
9402 reflections  
692 parameters  
H atoms treated by a mixture of independent and constrained refinement  
w = 1/[σ<sup>2</sup>(F<sub>o</sub><sup>2</sup>) + (0.0274P)<sup>2</sup> + 89.5191P]  
where P = (F<sub>o</sub><sup>2</sup> + 2F<sub>c</sub><sup>2</sup>)/3  
(Δ/σ)<sub>max</sub> = 0.001  
Δρ<sub>max</sub> = 1.37 e Å<sup>-3</sup>  
Δρ<sub>min</sub> = -0.72 e Å<sup>-3</sup>

**Table 1**  
Selected geometric parameters (Å, °).

Ru1—O1	2.204 (5)	Ru1—P46	2.3707 (17)
Ru1—P31	2.3254 (16)	Ru1—P16	2.4252 (17)
Ru1—P1	2.3659 (18)	Ru1—Cl1	2.4804 (15)
O1—Ru1—P31	91.90 (14)	P1—Ru1—P16	84.54 (6)
O1—Ru1—P1	169.20 (14)	P46—Ru1—P16	174.58 (5)
P31—Ru1—P1	97.82 (5)	O1—Ru1—Cl1	78.44 (14)
O1—Ru1—P46	90.15 (15)	P31—Ru1—Cl1	169.14 (5)
P31—Ru1—P46	83.89 (5)	P1—Ru1—Cl1	92.25 (5)
P1—Ru1—P46	95.55 (6)	P46—Ru1—Cl1	91.13 (5)
O1—Ru1—P16	88.92 (15)	P16—Ru1—Cl1	83.46 (5)
P31—Ru1—P16	101.47 (6)		

**Table 2**  
Hydrogen-bond geometry (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
O1—H2A...Cl1	0.91 (2)	2.47 (7)	2.970 (5)	115 (6)

The H atoms of the coordinated water molecule were located in a difference map; in the refinement their distance from atom O1, to which they are bound, was restrained to be 0.89 Å, with U<sub>iso</sub>(H) = 1.5U<sub>eq</sub>(O1). Atom O2 of the uncoordinated water molecule was refined at half occupancy, as the molecule was disordered around a twofold axis; in this case the H atoms were not located. C-bound H atoms were treated as riding [C—H = 0.95 and 0.99 Å and U<sub>iso</sub>(H) = 1.2U<sub>eq</sub>(C)]. One dichloromethane molecule was refined with full occupancy, but was disordered over two half-occupancy positions identified by C59A and C59B. All C—Cl bond distances in the molecule were restrained to be equal. The dichloromethane molecule containing C60 was refined with half occupancy, being disordered around a twofold axis; the two C—Cl bond distances were restrained to be the same as in the other dichloromethane molecule. Restraints for similarity and rigid-bond approximation were applied to all the Cl and C atoms, and the atoms of the anion and the uncoordinated solvent molecules were restrained to approximately isotropic behaviour. The highest residual electron-density peak is located close to the disordered atom Cl1B. The high value of R<sub>int</sub> is likely to be related to the disorder on the solvent molecules.

Data collection: COLLECT (Hooft, 1998); cell refinement: EVALCCD (Duisenberg *et al.*, 2003); data reduction: EVALCCD; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEPIII (Burnett & Johnson, 1996); software used to prepare material for publication: SHELXTL (Sheldrick, 2001) and CIFTAB (Sheldrick, 1993).

We thank the Academy of Finland for a research grant (KR and LR) and the Socrates/Erasmus Programme (JF and JR) for financial support. The Fundação para a Ciência e a Tecnologia (FCT, Portugal) is also gratefully acknowledged for partial support of this research through the project POCTI/CTM/41495/2001.

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