

Luca Russo,^a João Figueira,^b João Rodrigues^b and Kari Rissanen^{a*}^aNanoscience Centre, Department of Chemistry, University of Jyväskylä, PO Box 35, 40014 Jyväskylä, Finland, and ^bCentro de Química da Madeira, LQMM, Universidade da Madeira, 9000-390 Funchal, Portugal

Correspondence e-mail: krissane@cc.jyu.fi

Key indicators

Single-crystal X-ray study
 $T = 173$ K
Mean $\sigma(\text{C}-\text{C}) = 0.011$ 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>.***cis*-Aquabis[bis(diphenylphosphino)ethane- κ_2P,P']-chlororuthenium(II) hexafluorophosphate dichloromethane sesquisolvate hemihydrate**

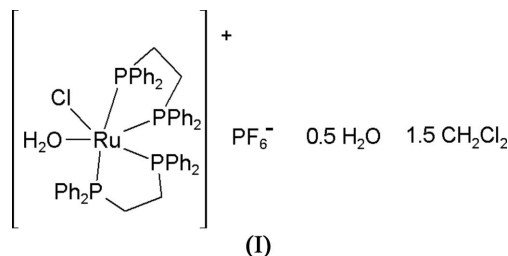
In the title compound, $[\text{RuCl}(\text{C}_{26}\text{H}_{24}\text{P})_2(\text{H}_2\text{O})]\text{PF}_6 \cdot 1.5\text{CH}_2\text{Cl}_2 \cdot 0.5\text{H}_2\text{O}$, the complex Ru^{II} cation is in a slightly distorted octahedral environment, chelated by two bis(diphenylphosphino)ethane ligands, 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*- $[\text{Ru}(\text{P}_2\text{C}_{26}\text{H}_{24})_2\text{Cl}_2]$ (0.280 g, 0.29 mmol), NH_4PF_6 (pre-dried under high vacuum, 0.082 g, 0.50 mmol) in pre-

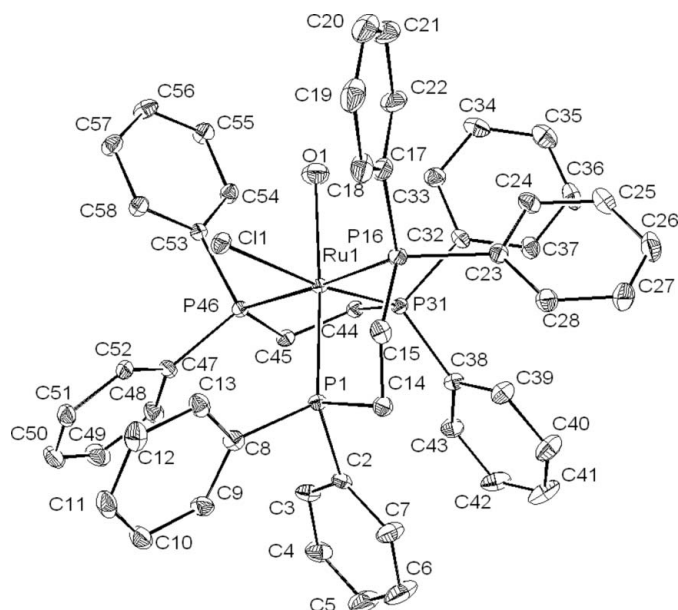


Figure 1
Structure of the complex cation showing the atom numbering scheme and with H atoms omitted. Displacement ellipsoids are drawn at the 30% probability level.

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(C₂₆H₂₄P)₂(H₂O)]PF₆·
1.5CH₂Cl₂·0.5H₂O
M_r = 1233.70
Monoclinic, *C*2/*c*
a = 37.707 (12) Å
b = 14.552 (3) Å
c = 25.583 (5) Å
β = 129.777 (19)°
V = 10789 (5) Å³
Z = 8

D_x = 1.519 Mg m⁻³
Mo *K*α radiation
Cell parameters from 8488
reflections
θ = 2.5–27.5°
μ = 0.70 mm⁻¹
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_{min} = 0.768, *T_{max}* = 0.973
66705 measured reflections
9402 independent reflections

6671 reflections with *I* > 2σ(*I*)
R_{int} = 0.129
θ_{max} = 25.0°
h = -44 → 44
k = -17 → 16
l = -30 → 30

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.067
wR(*F*²) = 0.140
S = 1.10
9402 reflections
692 parameters
H atoms treated by a mixture of
independent and constrained
refinement

w = 1/[σ²(*F_o*²) + (0.0274*P*)²
+ 89.5191*P*]
where *P* = (*F_o*² + 2*F_c*²)/3
(Δ/σ)_{max} = 0.001
Δρ_{max} = 1.37 e Å⁻³
Δρ_{min} = -0.72 e Å⁻³

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, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
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_{iso}*(H) = 1.5*U_{eq}*(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_{iso}*(H) = 1.2*U_{eq}*(C)]. One dichloromethane molecule was refined with full occupancy, but was disordered over two half-occupancy positions identified by C59*A* and C59*B*. 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 Cl1*B*. The high value of *R_{int}* 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.

References

- Burnett, M. H. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
Chang, C.-W., Ting, P.-C., Lin, Y.-C., Lee, G.-H. & Wang, Y. (1998). *J. Organomet. Chem.* **533**, 417–425.

- Chin, B., Lough, A. J., Morris, R. H., Schweitzer, C. T. & D'Agostino, C. (1994). *Inorg. Chem.* **33**, 6278–6288.
- Duisenberg, A. J. M., Kroon-Batenburg, L. M. J. & Shreurs, A. M. M. (2003). *J. Appl. Cryst.* **36**, 220–229.
- Fronczek, F. R., Breaux, H., McBride, T. G. & Srivastana, R. S. (2001) Private communication.
- Hooft, R. W. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
- Lobana, T. S., Singh, R. & Tiekink, E. R. T. (1990). *J. Coord. Chem.* **21**, 225–229.
- Polam, J. R. & Porter, L. C. (1993). *J. Coord. Chem.* **29**, 109–119.
- Sheldrick, G. M. (1993). *CIFTAB*. University of Göttingen, Germany. Modified by W. Clegg.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Sheldrick, G. M. (2001). *SHELXTL*. Version 6. Bruker AXS Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (2003). *SADABS*. University of Göttingen, Germany.
- Willis, A. C., Dalton, G. T., Morrall, J. P., Cifuentes, M. P. & Humphrey, M. G. (2004). *Acta Cryst.* **E60**, m1122–m1123.