

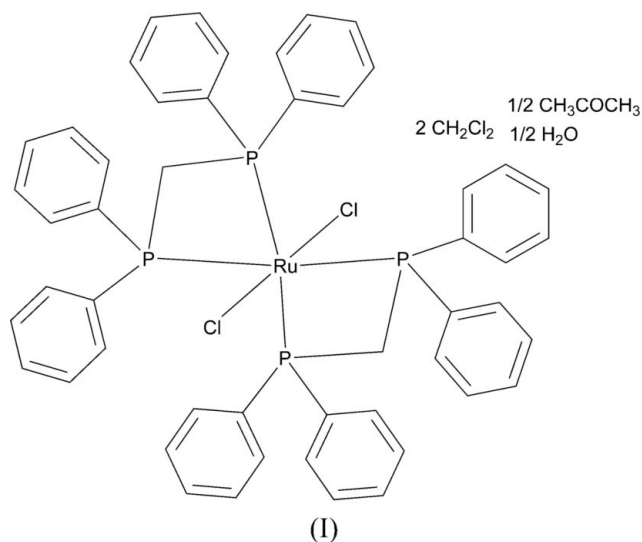
João Figueira,<sup>a</sup> João Rodrigues<sup>a\*</sup>  
and Kari Rissanen<sup>b</sup><sup>a</sup>Centro de Química da Madeira, LQCM/ MMRG, Departamento de Química da Universidade da Madeira, 9000-390 Funchal, Portugal, and <sup>b</sup>Nanoscience Center, Department of Chemistry, University of Jyväskylä, PO Box 35, 40014 Jyväskylä, Finland

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

## Key indicators

Single-crystal X-ray study  
 $T = 173$  K  
Mean  $\sigma(C-C) = 0.008$  Å  
Disorder in solvent or counterion  
 $R$  factor = 0.065  
 $wR$  factor = 0.166  
Data-to-parameter ratio = 19.0For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.***trans*-Bis[bis(diphenylphosphino)methane- $\kappa^2P,P'$ ]-dichlororuthenium(II) dichloromethane disolvate acetone hemisolvate hemihydrate**The title compound,  $[\text{RuCl}_2(\text{C}_{25}\text{H}_{22}\text{P}_2)_2] \cdot 2\text{CH}_2\text{Cl}_2 \cdot 0.5\text{C}_3\text{H}_6\text{O} \cdot 0.5\text{H}_2\text{O}$ , was obtained as unreacted starting material from our attempts to prepare acetylide complexes of ruthenium, based on 1,4-diethoxy-2,5-diethynylbenzene bridging ligands, in a route towards molecular wires. The complex is centrosymmetric.

## Comment

The *trans*- $[\text{RuCl}_2(\text{dppm})_2]$  complex [dppm is bis(diphenylphosphino)methane] is a useful starting material, commonly used for the preparation of mononuclear, as well as di- and polynuclear complexes (Faulkner *et al.*, 1994). The title compound, (I), has been characterized previously by Chatt & Hayter (1961) and Mague & Mitchener (1972). Later, Mason *et al.* (1976) obtained the same complex using a modified synthesis strategy and different starting materials. The *trans* chloride geometry was proved by spectroscopic techniques only, namely  $^{31}\text{P}$  NMR in solution. The chemical behaviour of the *cis*- and *trans*- $[\text{RuCl}_2(\text{dppm})_2]$  isomers has been thoroughly studied by Sullivan & Meyer (1982) and other authors (Zhu *et al.*, 1997; Higgins *et al.*, 2000). Four single-crystal structures containing the complex  $[\text{RuCl}_2(\text{dppm})_2]$  have been published; while three of them contain the *cis* isomer – the first as the non-solvated form (Chakravarty *et al.*, 1984), the second cocrystallized with the *fac*- $[\text{RuCl}_3(\text{dppm})(\text{NO})]$  complex (Batista *et al.*, 1999) and the third as a methanol solvate (Keller *et al.*, 2003) – only one has a non-solvated *trans* configuration (Chakravarty *et al.*, 1984). By recrystallization of *trans*- $[\text{RuCl}_2(\text{dppm})_2]$ , we obtained the title compound, (I).Received 24 October 2006  
Accepted 21 November 2006

In the crystal structure of (I), the Ru<sup>II</sup> atom (site symmetry  $\bar{1}$ ) has a distorted octahedral coordination *via* the four P atoms of the two chelating dppm ligands and the two chloride anions in *trans* geometry (Fig. 1 and Table 1). The dichloromethane (DCM) solvent molecules interact with the chloride anions *via* weak C—H...Cl<sup>−</sup> bonds.

## Experimental

Crystals of *trans*-[RuCl<sub>2</sub>(dppm)<sub>2</sub>]·2DCM were isolated as the unreacted product from the reaction between the unsolvated complex (0.665 g, 0.71 mmol), TIPF<sub>6</sub> (298 mg, 0.85 mmol) and 1,4-diethoxy-2,5-diethynylbenzene (51 mg, 0.24 mmol) in dried tetrahydrofuran (THF, 20 ml). After stirring for 6 d at room temperature under a nitrogen atmosphere, the solvent was removed under vacuum from the resulting dark-yellow solution. This yielded a dark-brown solid which was extracted with THF and then with dichloromethane (15 ml). To the latter fraction, triethylamine (3 ml, 21 mmol) was added and the mixture was stirred for a further 6 d, after which the resulting yellow mixture was filtered and evaporated under vacuum. The resulting yellow solid was washed with dry diethyl ether and *n*-hexane and then redissolved in dry CH<sub>2</sub>Cl<sub>2</sub> (10 ml). Acetone (5 ml) was then added and the mixture cooled to 253 K and held at that temperature for 7 d. The solution was filtered twice to remove a black oil that had formed and then more acetone (5 ml) was added. The mixture was allowed to stand for another 4 d at 253 K before it yielded bright-yellow crystals suitable for X-ray diffraction.

### Crystal data

[RuCl <sub>2</sub> (C <sub>25</sub> H <sub>22</sub> P <sub>2</sub> ) <sub>2</sub> ]·2CH <sub>2</sub> Cl <sub>2</sub> · 0.5C <sub>3</sub> H <sub>6</sub> O·0.5H <sub>2</sub> O	$\gamma = 105.397 (1)^\circ$
$M_r = 1170.61$	$V = 1326.79 (6) \text{ \AA}^3$
Triclinic, $P\bar{1}$	$Z = 1$
$a = 10.9227 (3) \text{ \AA}$	$D_x = 1.465 \text{ Mg m}^{-3}$
$b = 11.2603 (3) \text{ \AA}$	Mo $K\alpha$ radiation
$c = 11.6768 (2) \text{ \AA}$	$\mu = 0.76 \text{ mm}^{-1}$
$\alpha = 92.561 (2)^\circ$	$T = 173 (2) \text{ K}$
$\beta = 105.098 (1)^\circ$	Block, yellow
	$0.30 \times 0.30 \times 0.25 \text{ mm}$

### Data collection

Bruker–Nonius KappaCCD diffractometer	6011 independent reflections
$\varphi$ and $\omega$ scans	5247 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{\text{int}} = 0.029$
9274 measured reflections	$\theta_{\text{max}} = 27.4^\circ$

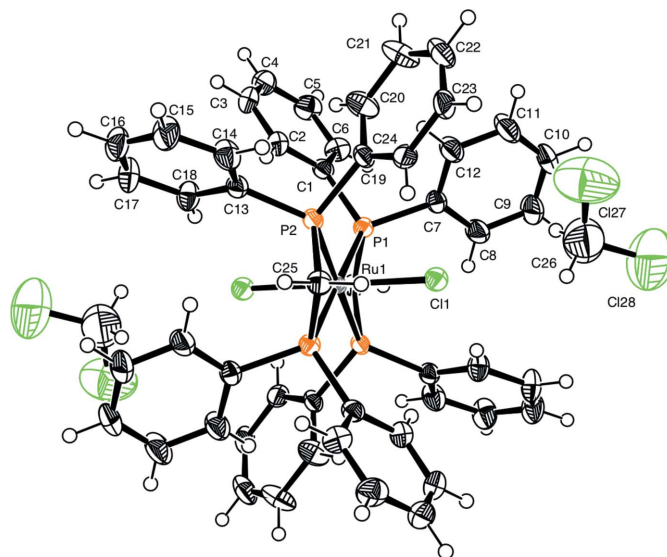
### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0603P)^2 + 6.4786P]$
$R[F^2 > 2\sigma(F^2)] = 0.066$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.166$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.09$	$\Delta\rho_{\text{max}} = 1.35 \text{ e \AA}^{-3}$
6011 reflections	$\Delta\rho_{\text{min}} = -1.10 \text{ e \AA}^{-3}$
316 parameters	
H-atom parameters constrained	

**Table 1**

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

Ru1—P2	2.3357 (11)	Ru1—Cl4	2.4285 (10)
Ru1—P3	2.3713 (11)		
P2—Ru1—P3	108.97 (4)	P3—Ru1—Cl4	80.81 (4)
P2—Ru1—Cl4	94.61 (4)		



**Figure 1**

The molecular structure showing the selected atom numbering and the solvent DCM molecules. Displacement ellipsoids are drawn at the 40% probability level.

The quite high residual electron density, not relating to the disolvated complex, was modelled as disordered acetone and water with partial occupancy ( $\frac{1}{2}$  and  $2 \times \frac{1}{4}$ , respectively). The remaining residual electron density ( $1.35 \text{ e \AA}^{-3}$ ) resides close to the DCM molecules and was not modelled. The deepest hole is located  $0.53 \text{ \AA}$  from atom Cl27.

Data collection: COLLECT (Hooft, 1998); cell refinement: SCALEPACK; data reduction: DENZO and SCALEPACK (Otwinowski & Minor, 1997); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999).

This research was supported by Fundação para a Ciência e a Tecnologia (Portugal) through FEDER-funded Project POCTI/CTM/41495/2001 (JF and JR) and by the sabbatical research grant SFRH/BSAB/632/2006. JR thanks the University of Jyväskylä for supporting his stay as a visiting professor at the Nanoscience Center, Department of Chemistry. The Academy of Finland is gratefully acknowledged for a research grant (No. 205729, to KR).

## References

- Batista, A. A., Pereira, C., Wohnrath, K., Queiroz, S. L., Santos, R. H. de A., Gambardella, M. T. & do, P. (1999). *Polyhedron*, **18**, 2079–2083.
- Chakravarty, A. R., Cotton, F. A. & Schwotzer, W. (1984). *Inorg. Chim. Acta*, **84**, 179–185.
- Chatt, J. & Hayter, R. G. (1961). *J. Chem. Soc.* pp. 896–904.
- Duisenberg, A. J. M., Kroon-Batenburg, L. M. J. & Schreurs, A. M. M. (2003). *J. Appl. Cryst.* **36**, 220–229.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Faulkner, C. W., Ingham, S. L., Khan, M. S., Lewis, J., Long, N. J. & Raithby, P. R. (1994). *J. Organomet. Chem.* **482**, 139–145.
- Higgins, S. J., Stuart, C. A. & Mills, A. (2000). *Inorg. Chem. Commun.* **3**, 208–210.

- Hooft, R. W. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
- Keller, A., Jasionka, B., Glowiak, T., Ershov, A. & Matusiak, R. (2003). *Inorg. Chim. Acta*, **344**, 49–60.
- Mague, J. T. & Mitchener, J. P. (1972). *Inorg. Chem.* **11**, 2714–2720.
- Mason, R., Meek, D. W. & Scollary, G. R. (1976). *Inorg. Chim. Acta*, **16**, L11–L12.
- Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Sullivan, B. P. & Meyer, T. J. (1982). *Inorg. Chem.* **21**, 1037–1040.
- Zhu, Y., Wolf, M. O. & Yap, G. P. A. (1997). *Inorg. Chem.* **36**, 5483–5487.