

metal-organic papers

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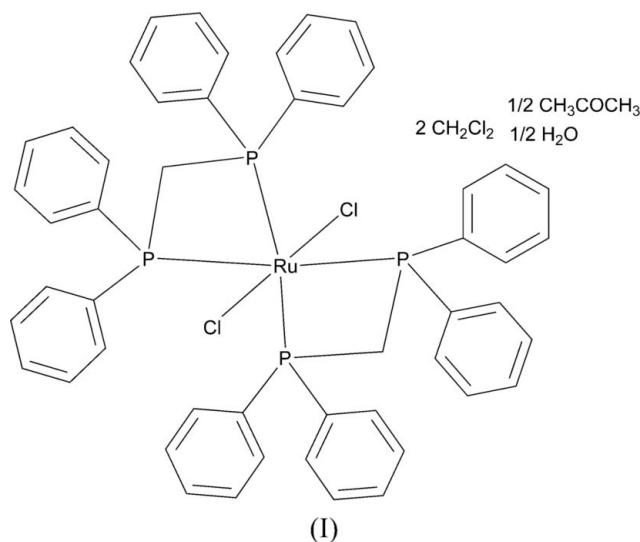
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Key indicators

Single-crystal X-ray study
 $T = 173\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.008\text{ \AA}$
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- κ^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.Received 24 October 2006
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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}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).

In the crystal structure of (I), the Ru^{II} 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[−] bonds.

Experimental

Crystals of *trans*-[RuCl₂(dppm)₂]·2DCM were isolated as the unreacted product from the reaction between the unsolvated complex (0.665 g, 0.71 mmol), TIPF₆ (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 CH₂Cl₂ (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 ₂ (C ₂₅ H ₂₂ P ₂) ₂]·2CH ₂ Cl ₂ · 0.5C ₃ H ₆ O·0.5H ₂ 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
φ and ω 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 (\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)		

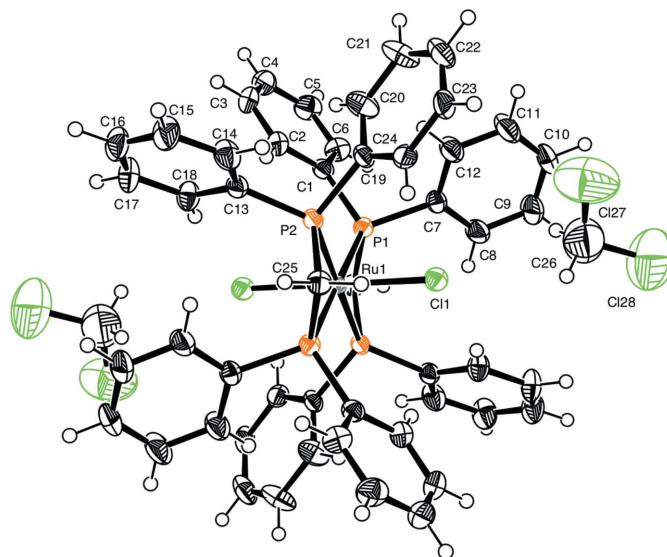


Figure 1

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 e \AA^{-3}) resides close to the DCM molecules and was not modelled. The deepest hole is located 0.53 \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).

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