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trans-Bis[bis(diphenylphosphino)methane-κ^2P,P']-dichlororuthenium(II) dichloromethane disolvate acetone hemisolvate hemihydrate

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.008 Å
Disorder in solvent or counterion
R factor = 0.065
wR factor = 0.166
Data-to-parameter ratio = 19.0

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

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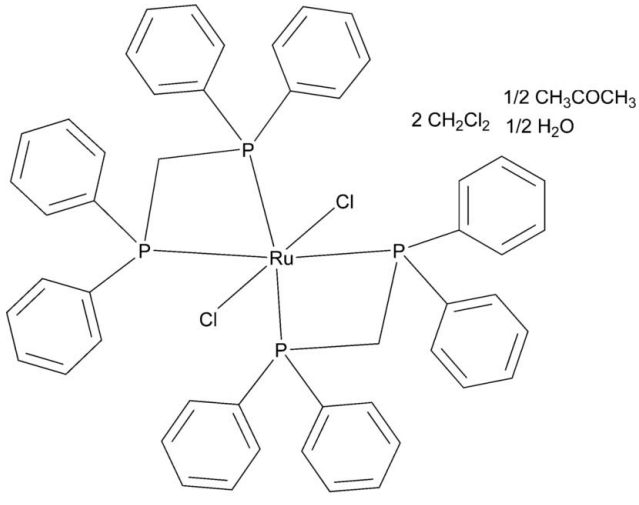
The title compound, $[RuCl_2(C_{25}H_{22}P_2)_2]\cdot 2CH_2Cl_2\cdot 0.5C_3H_6O\cdot 0.5H_2O$, was obtained as unreacted starting material from our attempts to prepare acetylide complexes of ruthenium, based on 1,4-diethoxy-2,5-diethynylbenzene bridging ligands, in a route towards molecular wires. The complex is centrosymmetric.

Comment

The *trans*-$[RuCl_2(dppm)_2]$ 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*-$[RuCl_2(dppm)_2]$ 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 $[RuCl_2(dppm)_2]$ 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*-$[RuCl_3(dppm)(NO)]$ 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*-$[RuCl_2(dppm)_2]$, we obtained the title compound, (I).



(I)

In the crystal structure of (I), the Ru^{II} atom (site symmetry $\overline{1}$) 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*-$[RuCl_2(dppm)_2]\cdot 2DCM$ were isolated as the unreacted product from the reaction between the unsolvated complex (0.665 g, 0.71 mmol), $TlPF_6$ (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_2Cl_2 (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

$[RuCl_2(C_{25}H_{22}P_2)_2]\cdot 2CH_2Cl_2\cdot 0.5C_3H_6O\cdot 0.5H_2O$	$\gamma = 105.397\ (1)^\circ$
$M_r = 1170.61$	$V = 1326.79\ (6)$ Å^3
Triclinic, $P\overline{1}$	$Z = 1$
$a = 10.9227\ (3)$ Å	$D_x = 1.465$ Mg m^{-3}
$b = 11.2603\ (3)$ Å	Mo $K\alpha$ radiation
$c = 11.6768\ (2)$ Å	$\mu = 0.76$ mm^{-1}
$\alpha = 92.561\ (2)^\circ$	$T = 173\ (2)$ K
$\beta = 105.098\ (1)^\circ$	Block, yellow
	0.30 × 0.30 × 0.25 mm

Data collection

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

Refinement

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

Table 1
Selected geometric parameters (Å, °).

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)		

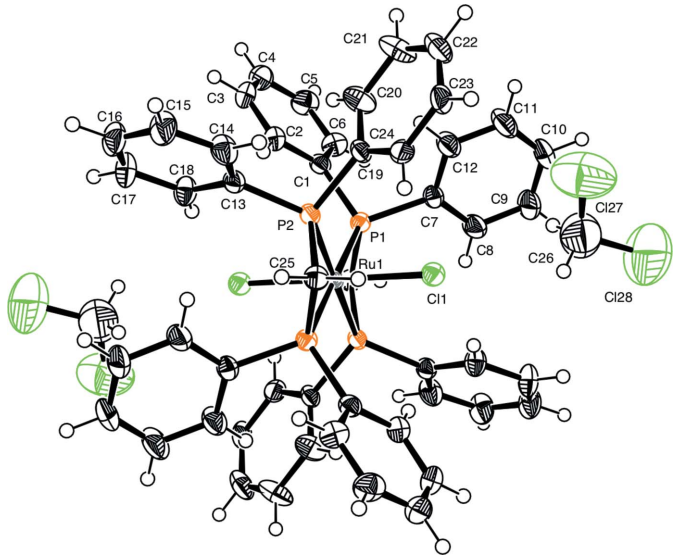


Figure 1
The molecular structure showing the selected atom numbering and the solvent DCM molecules. Displacement ellipsoids are drawn at the 40% probability level.

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 × $\frac{1}{4}$, respectively). The remaining residual electron density (1.35 e Å^{-3}) resides close to the DCM molecules and was not modelled. The deepest hole is located 0.53 Å 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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