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cis-[Bis(diphenylphosphino)ethane-κ^2P,P']-dichlororuthenium(II) dichloromethane disolvate

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The title compound, *cis*-$[RuCl_2(C_{26}H_{24}P_2)_2]\cdot 2CH_2Cl_2$, was obtained as an unexpected product from our attempts to prepare new ruthenium molecular wires using organic bridging ligands. Three solvates and a solvent-free structure of the isomeric complex with the chloride anions in a *trans* geometry have already been reported, while the *cis* isomer has been described only in solution studies prior to this work.

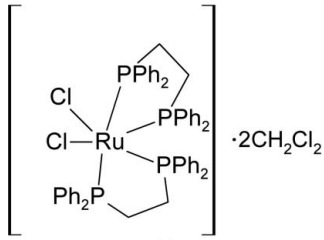
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.008 Å
Disorder in solvent or counterion
R factor = 0.071
wR factor = 0.182
Data-to-parameter ratio = 18.6

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

Comment

$[RuCl_2(dppe)_2]$ [dppe is (diphenylphosphino)ethane] was prepared for the first time as the *trans* isomer (Chatt & Hayter, 1961) and, more recently, the *cis* isomeric form was also isolated (Bautista *et al.*, 1991). The *cis*-$[RuCl_2(dppe)_2]$ complex is a useful starting material, commonly used for the preparation of mononuclear, as well as bi- and trinuclear, complexes (Lavastre *et al.*, 1997; Rigaut *et al.*, 2003), but its crystal structure has not been reported prior to this work. Four structures containing the complex *trans*-$[RuCl_2(dppe)_2]$ can be found in the literature, namely in the form of a dichloromethane solvate (Lobana *et al.*, 1990), a chloroform solvate (Fronczek *et al.*, 2001), a tetrahydrofuran solvate (Chang *et al.*, 1998) and a solvent-free structure (Polam & Porter, 1993). In the title compound, *cis*-$[RuCl_2(P_2C_{26}H_{24})_2]\cdot 2CH_2Cl_2$, (I), the complex Ru^{II} cation is in a slightly distorted octahedral environment, chelated by two dppe ligands, with two chloride anions in a mutually *cis* geometry completing the coordination environment (Fig. 1 and Table 1). In addition to the neutral complex, the asymmetric unit also contains two dichloromethane solvent molecules.



(I)

Experimental

The reaction was carried out in an oven-dried, vacuum/nitrogen cycled Schlenk flask, under a nitrogen atmosphere. *cis*-$[RuCl_2(dppe)_2]$ (0.283 g, 0.29 mmol) and 1,4-diethoxy-2,5-diethynylbenzene (0.023 g, 0.11 mmol) were dissolved in dry tetrahydrofuran (30 ml) in the presence of an excess of triethylamine (10 ml); after 24 h stirring at room temperature a dark-yellow suspension was

obtained. The crude yellow product, obtained upon removal from a light-red solution, was dried, washed with dry Et_2O (2 × 5 ml) and *n*-hexane (5 ml), and dried again under reduced pressure. At this point the product contained a mixture of the *cis*- and *trans*-$[RuCl_2(dppe)_2]$ complexes in an approximate ratio of 46:54 (based on ^{31}P NMR). Recrystallization of the product from CH_2Cl_2/Et_2O (3:3), at 253 K, gave a dark-orange powder (fraction *A*) and, mostly, very fine yellow needles (fraction *B*). Further recrystallization of fraction *B*, under the same conditions, gave yellow thick needles suitable for single-crystal X-ray diffraction analysis.

Crystal data

$[RuCl_2(C_{26}H_{24}P_2)_2]\cdot 2CH_2Cl_2$
M_r = 1138.61
Monoclinic, $P2_1/n$
a = 17.054 (3) Å
b = 13.182 (3) Å
c = 23.592 (5) Å
β = 103.05 (3)°
V = 5167 (2) Å^3
Z = 4
D_x = 1.464 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.77 mm^{-1}
T = 173 (2) K
Block, yellow
0.20 × 0.15 × 0.15 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.861, T_{max} = 0.893
29691 measured reflections
11446 independent reflections
7242 reflections with $I > 2\sigma(I)$
R_{int} = 0.100
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.071
$wR(F^2)$ = 0.182
S = 1.04
11446 reflections
614 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0675P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.19 e Å^{-3}
$\Delta\rho_{min}$ = −1.22 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ru1—P1	2.3825 (14)	Ru1—P16	2.3301 (14)
Ru1—Cl1	2.4710 (14)	Ru1—P31	2.3915 (14)
Ru1—Cl2	2.4752 (13)	Ru1—P46	2.3568 (14)
P1—Ru1—Cl1	88.48 (5)	Cl1—Ru1—P46	166.84 (4)
P1—Ru1—Cl2	86.63 (5)	Cl2—Ru1—P16	171.11 (5)
P1—Ru1—P16	84.50 (5)	Cl2—Ru1—P31	83.28 (5)
P1—Ru1—P31	167.19 (5)	Cl2—Ru1—P46	90.35 (5)
P1—Ru1—P46	104.41 (5)	P16—Ru1—P31	105.41 (5)
Cl1—Ru1—Cl2	87.71 (4)	P16—Ru1—P46	92.42 (5)
Cl1—Ru1—P16	91.46 (5)	P31—Ru1—P46	83.58 (5)
Cl1—Ru1—P31	83.26 (5)		

One dichloromethane molecule was refined as disordered over two positions, identified by C62*A* and C62*B*, Cl5*A* and C15*B*, Cl6*A* and C16*B*, and H62*A*–H62*D*, with occupancies of 0.552 (8) and 0.448 (8), respectively. All H atoms were refined using a riding model with C—H = 0.95 Å for aromatic atoms, C—H = 0.99 Å for CH_2 and $U_{iso}(H) = 1.2U_{eq}(C)$. The maximum and minimum electron-density peaks in the final difference map lie 0.93 Å from atom Ru1 and 0.79 Å from atom Ru1, respectively.

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).

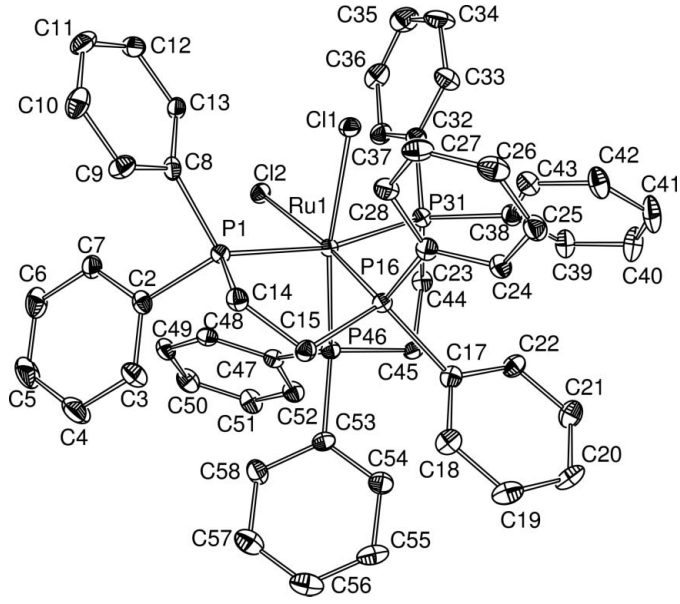


Figure 1
Plot of the complex (H atoms omitted), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms and solvent molecules have been omitted.

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