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Hydridodichlorotris(methyldiphenylphosphine)-rhodium(III) dichloromethane trisolvate

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Key indicators

Single-crystal X-ray study
T = 160 K
Mean σ(C–C) = 0.006 Å
R factor = 0.044
wR factor = 0.118
Data-to-parameter ratio = 21.0

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

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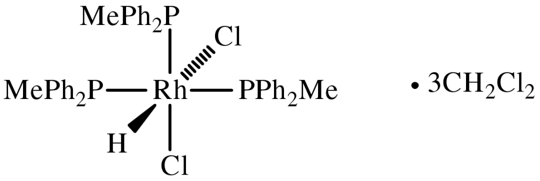
The title complex, $[RhHCl_2(PMePh_2)_3]\cdot 3CH_2Cl_2$, has octahedral coordination for Rh, with the three phosphine ligands arranged *mer* and the two chloro ligands mutually *cis*. Coordination bond lengths are in accord with the relative *trans* influence of the ligands.

Comment

The title compound, (I), was obtained as a minor product during a study of diboration reactions catalysed by metal complexes. It consists of a discrete neutral mononuclear complex of Rh^{III}, solvated in the crystal structure by three molecules of dichloromethane per molecule of complex. There are no special intermolecular interactions.



(I)

Three phosphine ligands are arranged in a *mer* configuration, while the two chloro ligands are mutually *cis*. This arrangement places one chloro ligand opposite the central phosphine, while the other lies opposite the hydrido ligand (Fig. 1). The variation in rhodium-ligand bond lengths (Table 1) is in accord with the relative strength of the *trans*-influence of the three types of ligand: hydrido > phosphine > chloro. Thus, the shortest Rh—P bond is to P2, opposite Cl2, while Rh—Cl1, *trans* to H1, is markedly longer than Rh—Cl2. Angular distortions from ideal octahedral geometry are mainly a consequence of the steric bulk of the phosphine ligands and the small size of the hydrido ligand.

A search of the Cambridge Structural Database (Allen & Kennard, 1993; April 2002 Release) shows that a *mer* configuration (six entries) is more common than a *fac* configuration (only one entry) for octahedral complexes of Rh with three phosphine ligands. The freely refined Rh—H1 bond length of 1.54 (4) Å is essentially the same as the mean value for 160 occurrences in 123 structures in the Database.

Experimental

$[RhCl(PPh_3)_3]$ (Wilkinson's catalyst; 0.074 g, 0.08 mmol) and $B_2(1,2\text{–}O_2\text{-}4\text{–}MeC_6H_3)_2$ (0.032 g, 0.12 mmol) were dissolved together in CH_2Cl_2 (3 ml) in order to form $[RhCl(PPh_3)_2\{B(1,2\text{-}O_2\text{-}4\text{-}MeC_6H_3)\}_2]$ (Clegg *et al.*, 1994), and the solution was stirred rapidly for *ca* 20 min.

To the resulting yellow solution was added $PMePh_2$ (0.045 ml, 0.048 g, 0.24 mmol), resulting in a change of colour from yellow to orange. The reaction mixture was layered with hexanes (8 ml) and kept at 243 K for several days, during which solvent diffusion yielded colourless crystals of the title complex as a minor product.

Crystal data

$[RhHCl_2(C_{13}H_{13}P)_3]\cdot 3CH_2Cl_2$
$M_r = 1030.21$
Monoclinic, $P2_1/n$
$a = 12.5494$ (8) Å
$b = 18.3233$ (12) Å
$c = 20.0687$ (13) Å
$\beta = 92.063$ (2)°
$V = 4611.7$ (5) Å^3
$Z = 4$

$D_x = 1.484$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 16248 reflections
$\theta = 2.2$–28.4°
$\mu = 0.97$ mm^{-1}
$T = 160$ (2) K
Block, colourless
0.50 × 0.32 × 0.30 mm

Data collection

Siemens SMART CCD diffractometer
ω rotation with narrow frames
Absorption correction: multi-scan (*XPREP* in *SHELXTL*; Sheldrick, 1997)
$T_{\min} = 0.642$, $T_{\max} = 0.876$
27485 measured reflections

10376 independent reflections
8288 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.041$
$\theta_{\max} = 28.4°$
$h = -16 \rightarrow 11$
$k = -23 \rightarrow 24$
$l = -26 \rightarrow 26$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.118$
$S = 1.11$
10376 reflections
493 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0449P)^2 + 10.9692P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 1.02$ e Å^{-3}
$\Delta\rho_{\min} = -1.51$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Rh—H1	1.54 (4)	Rh—P1	2.3585 (9)
Rh—Cl1	2.5107 (8)	Rh—P2	2.2577 (9)
Rh—Cl2	2.4260 (8)	Rh—P3	2.3518 (9)
H1—Rh—Cl1	176.3 (14)	Cl1—Rh—P3	88.45 (3)
H1—Rh—Cl2	86.8 (14)	Cl2—Rh—P1	83.91 (3)
H1—Rh—P1	86.6 (14)	Cl2—Rh—P2	168.58 (3)
H1—Rh—P2	81.9 (14)	Cl2—Rh—P3	84.78 (3)
H1—Rh—P3	95.2 (14)	P1—Rh—P2	97.13 (3)
Cl1—Rh—Cl2	92.75 (3)	P1—Rh—P3	168.43 (3)
Cl1—Rh—P1	89.66 (3)	P2—Rh—P3	94.44 (3)
Cl1—Rh—P2	98.61 (3)		

The hydride ligand was located in a difference map and its positional parameters were refined freely; the U value was fixed at 0.025 Å^2, comparable to the $U_{\rm eq}$ values of the other atoms bonded to Rh. Other H atoms were placed geometrically and refined with a riding model (including free rotation about C—C bonds), and with $U_{\rm iso}$ constrained to be 1.2 (1.5 for methyl groups) times $U_{\rm eq}$ of the carrier atom. The largest residual electron-density features lie close to one of the solvent molecules, for which atom C42 has highly anisotropic displacement parameters and the two C—Cl bonds deviate markedly from normal values; disorder is likely for this molecule, but it was not resolved.

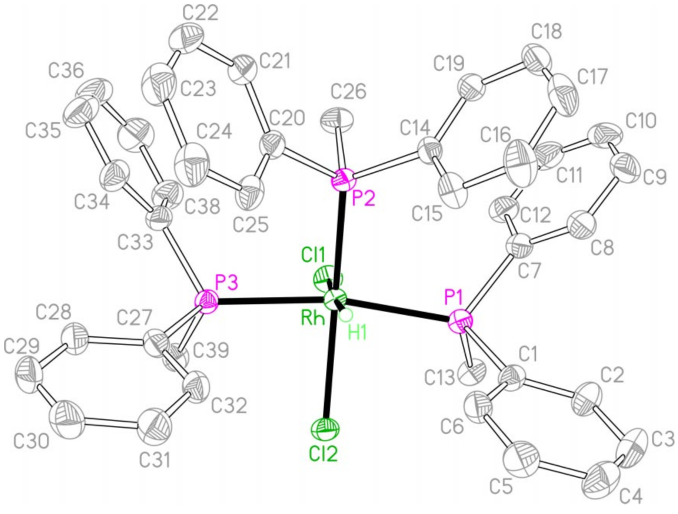


Figure 1
The molecular structure, with atom labels and 50% probability ellipsoids for non-H atoms. Solvent molecules and H atoms bonded to carbon have been omitted.

Data collection: *SMART* (Siemens, 1994); cell refinement: local programs; data reduction: *SAINT* (Siemens, 1994); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and local programs.

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