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Chloro-*cis*-dihydro-*mer*-tris(triphenylphosphine)-rhodium(III) dichloromethane hemisolvate

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The title complex, $[RhClH_2(C_{18}H_{15}P)_3]\cdot0.5CH_2Cl_2$, has distorted octahedral coordination of Rh as a consequence of the bulk of the phosphine ligands and the relatively small size of hydride ligands; the latter were not directly located in the crystallographic analysis. The dichloromethane solvent molecule is disordered.

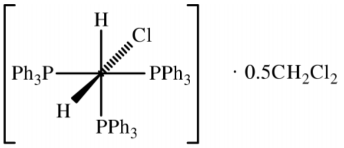
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.008 Å
H-atom completeness 96%
Disorder in solvent or counterion
R factor = 0.038
wR factor = 0.109
Data-to-parameter ratio = 10.9

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

Comment

The crystal structure of the tetrahydrofuran solvate of the title complex, (I), has been reported previously (Burgess *et al.*, 1992). The complex has essentially the same structure in the dichloromethane hemisolvate described here (Fig. 1).



(I)

Although the hydride ligands could not be unambiguously located in a difference map, it is clear from the positions of the other ligands that they are mutually *cis*, one of them lying opposite the chloro ligand and the other opposite the central triphosphine ligand out of the meridionally disposed set of three. The high *trans* influence of hydride is reflected in the relatively long Rh—Cl bond and in the lengthening of Rh—P2 relative to Rh—P1 and Rh—P3 (Table 1).

Deviations from ideal octahedral geometry at Rh are marked, and are due to the bulk of the phosphine ligands and the small size of the hydride ligands. Thus, the angles P2—Rh—P1, P2—Rh—P3 and P2—Rh—Cl are considerably greater than 90°.

Experimental

The title complex was prepared from Wilkinson's catalyst and molecular hydrogen, according to Burgess *et al.* (1992), and was crystallized from dichloromethane.

Crystal data

$[RhClH_2(C_{18}H_{15}P)_3]\cdot0.5CH_2Cl_2$
$M_r = 969.6$
Triclinic, $P\bar{1}$
a = 9.390 (5) Å
b = 12.581 (6) Å
c = 20.096 (10) Å
α = 83.30 (5)°
β = 83.42 (4)°
γ = 76.64 (4)°
V = 2285 (2) Å^3
Z = 2
D_x = 1.409 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 31 reflections
θ = 10.2–12.5°
μ = 0.63 mm^{-1}
T = 298 (2) K
Block, yellow
0.23 × 0.20 × 0.13 mm

Data collection

Stoe–Siemens diffractometer
ω/θ scans
Absorption correction: ψ scan (*XPREP* in *SHELXTL*; Sheldrick, 1997)
$T_{\rm min} = 0.86$, $T_{\rm max} = 0.96$
6142 measured reflections
5972 independent reflections
4522 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.057$
$\theta_{\rm max} = 22.5°$
$h = -9 \rightarrow 10$
$k = -13 \rightarrow 13$
$l = -11 \rightarrow 21$
5 standard reflections
frequency: 60 min
intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.109$
$S = 1.06$
5972 reflections
550 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0655P)^2 + 1.2789P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 1.50$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.68$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Rh—Cl1	2.4714 (18)	Rh—P2	2.4411 (18)
Rh—P1	2.3256 (16)	Rh—P3	2.3186 (16)
Cl1—Rh—P1	83.10 (6)	P1—Rh—P2	103.12 (6)
Cl1—Rh—P2	105.29 (6)	P1—Rh—P3	154.79 (5)
Cl1—Rh—P3	85.11 (6)	P2—Rh—P3	101.46 (6)

Data above $\theta = 22.5°$ were weak and the data collection was limited to this maximum value. Phosphine-ligand and solvent H atoms were placed geometrically and refined with a riding model, and with $U_{\rm iso}$ constrained to be 1.2 times $U_{\rm eq}$ of the carrier atom. The hydride ligands could not be located reliably in a difference map, and they were not included. There are two residual electron-density peaks with heights 1.50 and 1.35 e Å^{-3}, both about 1.1 Å from Rh, but not in appropriate positions for the hydride ligands; all other features in the final difference map are insignificant. The solvent molecule is disordered over an inversion centre, with common sites for the chlorine atoms and distinct half-occupancy sites for C and H atoms.

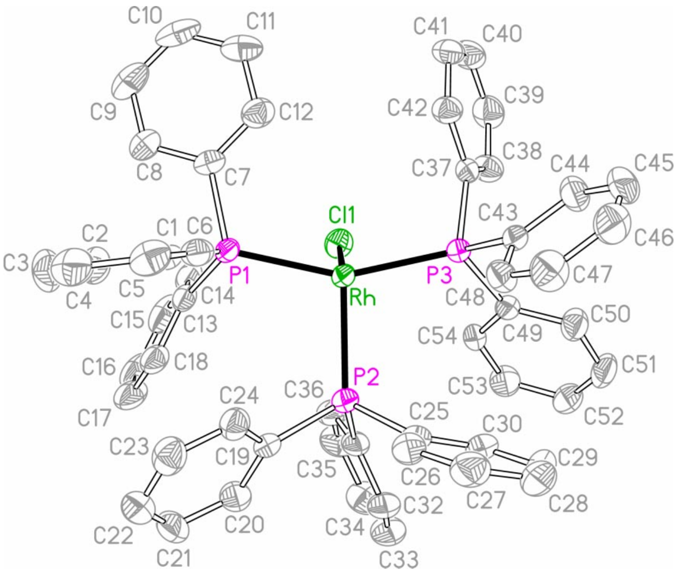


Figure 1
The molecular structure of (I), with atom labels and 50% probability ellipsoids for non-H atoms. The solvent molecule has been omitted.

Data collection: *DIF*4 (Stoe & Cie, 1988); cell refinement: *DIF*4; data reduction: local programs; 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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