

Chloro(methyl)(η^5 -pentamethylcyclopentadienyl)-(trimethylphosphine)rhodium(III)

Ola F. Wendt

Inorganic Chemistry, Department of Chemistry,
Lund University, Box 124, S-221 00 Lund,
Sweden

Correspondence e-mail: ola.wendt@inorg.lu.se

Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.008\text{ \AA}$

R factor = 0.054

wR factor = 0.165

Data-to-parameter ratio = 35.8

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

The title compound, $\text{RhCp}^*\text{Me}(\text{Cl})(\text{PMe}_3)$ or $[\text{RhCl}(\text{CH}_3)(\text{C}_{10}\text{H}_{15})(\text{C}_3\text{H}_9\text{P})]$, where Cp^* is pentamethylcyclopentadienyl, has a pseudo-tetrahedral coordination geometry. The Cp^* ligand is planar and η^5 -coordinated. The molecular structure can be described as a so-called three-legged piano-stool. The rhodium–methyl, rhodium–chlorine and rhodium–phosphorus distances are 2.255 (4), 2.3764 (18) and 2.2436 (13) Å, respectively.

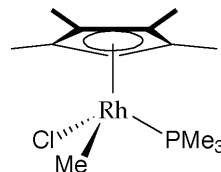
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Comment

The title compound, (I) (Fig. 1), was first prepared by Jones & Feher (1984) and belongs to a group of complexes that have been used extensively in the activation of small molecules (Lefort *et al.*, 1998; Arndtsen *et al.*, 1995). However, there are no reported crystal structures of chloro–methyl complexes of this type.



The coordination geometry around rhodium is pseudo-tetrahedral and the Cp^* ligand is planar and η^5 -coordinated. This gives rise to a three-legged piano-stool where the angles around rhodium involving the monodentate ligands are

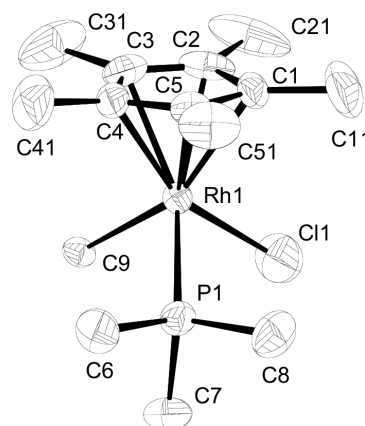


Figure 1

Numbering scheme with displacement ellipsoids (30% probability) for the title compound.

around 88°. The deviation from idealized tetrahedral angles is probably dictated by the bulky Cp* ligand. Selected bond lengths and angles are given in Table 1. The closest contact between complexes is 2.04 (1) Å and is found between H9A and H9A(-x, -y, 1 - z). This short distance is probably an artefact that arises from the fact that H atoms are placed only to minimize intramolecular interactions. The C9...C9(-x, -y, 1 - z) distance is 3.870 (6) Å and rotation around the Rh—C9 bond makes the C—H distances much longer.

Experimental

RhCp*Me₂(PMe₃) was synthesized according to Jones & Feher (1984) and recrystallization from petroleum ether afforded red crystals of the title compound as a by-product.

Crystal data

[RhCl(CH₃)(C₁₀H₁₅)(C₃H₉P)]
M_r = 364.69
 Orthorhombic, *Pbca*
a = 8.9295 (18) Å
b = 14.792 (3) Å
c = 25.812 (5) Å
V = 3409.2 (12) Å³
Z = 8
D_x = 1.421 Mg m⁻³

Mo *K*α radiation
 Cell parameters from 6294 reflections
 $\theta = 2.8\text{--}25.4^\circ$
 $\mu = 1.23 \text{ mm}^{-1}$
T = 293 (2) K
 Prism, red
 0.15 × 0.03 × 0.03 mm

Data collection

Bruker SMART CCD diffractometer
 ω scans
 Absorption correction: empirical (SADABS; Sheldrick, 1996)
T_{min} = 0.789, *T_{max}* = 0.943
 33 212 measured reflections
 5506 independent reflections

2862 reflections with *I* > 2σ(*I*)
R_{int} = 0.069
 $\theta_{\text{max}} = 31.9^\circ$
h = -12 → 13
k = -20 → 21
l = -36 → 37
 Intensity decay: none

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.054
wR(*F*²) = 0.165
S = 1.00
 5506 reflections
 154 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0873P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.94 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -1.36 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

Rh1—C4	2.179 (4)	P1—C8	1.807 (6)
Rh1—C5	2.225 (4)	P1—C7	1.822 (6)
Rh1—C3	2.225 (5)	P1—C6	1.823 (5)
Rh1—C2	2.228 (5)	C1—C5	1.415 (6)
Rh1—C1	2.230 (4)	C1—C2	1.453 (8)
Rh1—P1	2.2436 (13)	C2—C3	1.344 (8)
Rh1—C9	2.255 (4)	C3—C4	1.419 (9)
Rh1—Cl1	2.3764 (18)	C4—C5	1.393 (7)
P1—Rh1—C9	89.02 (11)	C7—P1—Rh1	116.3 (2)
P1—Rh1—Cl1	87.88 (6)	C6—P1—Rh1	116.97 (19)
C9—Rh1—Cl1	87.55 (12)	C5—C1—C2	105.1 (4)
C8—P1—C7	103.3 (3)	C3—C2—C1	109.3 (5)
C8—P1—C6	103.2 (3)	C2—C3—C4	109.0 (5)
C7—P1—C6	102.7 (3)	C5—C4—C3	107.4 (5)
C8—P1—Rh1	112.6 (2)	C4—C5—C1	109.0 (4)

The highest residual electron density is located within 0.5 Å of C9 and Cl1.

Data collection: *SMART* (Bruker, 1995); cell refinement: *SAINT* (Bruker, 1995); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXL97*.

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