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trans-Carbonylchlorobis[diphenyl(4-tolyl)-phosphine]rhodium(I)

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The title compound, $[RhCl(C_{19}H_{17}P)_2(CO)]$, can be characterized as a Vaska-type complex containing a diphenyl(4-tolyl)phosphine ligand. The rhodium(I) metal centre has a square-planar coordination. The molecule is disordered, with the Rh atom lying on an inversion centre. The most important bond lengths and angles include Rh—P = 2.3315 (9) Å, Rh—Cl(*trans* CO) = 2.405 (2) Å, Rh—C(carbonyl) = 1.724 (11) Å and Rh—C—O = 178.0 (6)°.

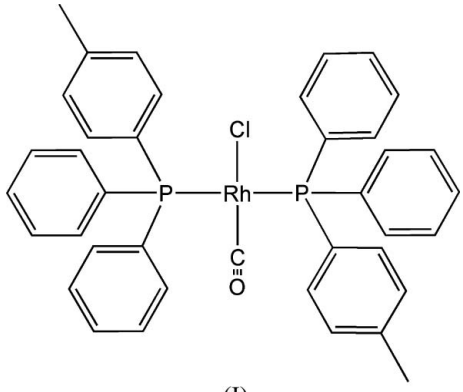
Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.024
wR factor = 0.062
Data-to-parameter ratio = 18.4

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

Comment

Rhodium(I) Vaska-type compounds containing chlorine and triphenylphosphine (PPh_3) have been extensively investigated in the past few decades (Ceriotti *et al.*, 1983; Chen *et al.*, 1991; Del Pra *et al.*, 1979; Muir & Ibers, 1969; Otto *et al.*, 1999). Angoletta (1959) reported the original Vaska complex, $[IrCl(PPh_3)_2(CO)]$, in 1959, but it was Vaska & Di Luzio (1961) who later correctly formulated the complex. Functionalization of one of the phenyl rings of PPh_3 with various substituents has also been studied: 4-Me (Suomalainen *et al.*, 2001), 4-Me-benzoate (Dutta *et al.*, 2003), 4-MeO (Suomalainen *et al.*, 2000). In this paper, the crystal structure of the title compound, (I), a 4-TolPh_2P derivative, is presented.



(I)

Compound (I) crystallizes with a square-planar geometry about the Rh^I metal centre (Fig. 1). Selected bond lengths and angles are given in Table 1. The Rh atom lies on a centre of symmetry, with the Cl atom and carbonyl unit disordered with a ratio of 0.5:0.5. Further disorder in the solid state is observed for the H atoms of the 4-methyl group.

The Rh—P, Rh—C and Rh—Cl bond distances are comparable with reported monofunctionalized phenyl–diphenylphosphine complexes (Table 2). The Rh—P bond in (I) is relatively long compared to those in similar structures (CSD, Version 5.27; Allen, 2002), but it correlates well with the tris-4-tolyl derivative (Otto *et al.*, 1999; Table 2). The short

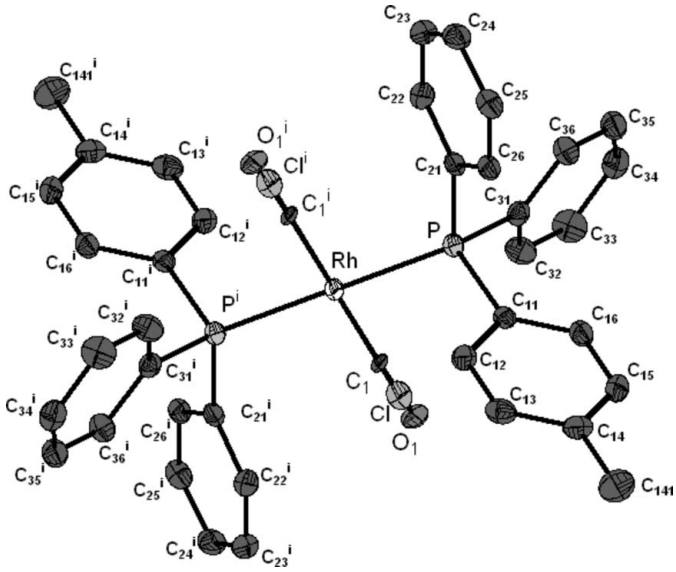


Figure 1
A sketch of the title compound (I), showing the disorder of the carbonyl group and chlorine atom. Displacement ellipsoids are drawn at the 50% probability level. For the carbon rings the first digit refers to ring number and the second digit to the atom in the ring. H atoms have been omitted for clarity [symmetry code: (i) $-x,-y,-z$].

intermolecular O1···H23^{i} contact of 2.644 (13) Å [symmetry code: (i) x, $1+y$, z] is an indication of the efficient packing mode in the crystal structure. There is no clear correlation between the functionalization of the phosphine ligand and the disordered packing mode of the carbonyl unit and the chlorine atom in the solid state. This can be observed from the fact that the triphenylphosphine complex and the title compound, (I), are disordered; however this disorder does not exist for the tri-4-tolylphosphine nor the other *ortho*-functionalized triphenylphosphine derivatives (Table 2).

Experimental

The title complex was synthesized by the addition of 4-TolPh$_2$P (42.5 mg, 0.154 mmol) to a 5 ml acetone solution of [Rh(μ-Cl)-(CO)$_2$]$_2$ (14.6 mg, 0.037 mmol). Slow evaporation of the solvent gave yellow crystals of the desired product suitable for X-ray diffraction (yield 46 mg, 85%).

Crystal data

[RhCl($C_{19}H_{17}P$)$_2$(CO)]
M_r = 718.96
Triclinic, $P\overline{1}$
a = 9.946 (5) Å
b = 9.969 (5) Å
c = 10.216 (5) Å
α = 87.813 (5)°
β = 68.850 (5)°
γ = 61.301 (5)°
V = 817.3 (7) Å^3
Z = 1
D_x = 1.461 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.73 mm^{-1}
T = 100 (2) K
Block, yellow
0.23 × 0.11 × 0.08 mm

Data collection

Bruker X8 APEX-II diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.850, T_{max} = 0.944
16054 measured reflections
3931 independent reflections
3648 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 28.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.062
S = 1.05
3931 reflections
214 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0268P)^2 + 0.5271P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Rh—C1	1.795 (5)	P—C21	1.8259 (18)
Rh—P	2.3315 (9)	P—C31	1.8328 (19)
Rh—Cl	2.397 (2)	C1—O1	1.117 (5)
P—C11	1.8194 (18)		
C1—Rh—P	87.41 (16)	C1^{i}—Rh—Cl	177.23 (17)
C1^{i}—Rh—P	92.59 (16)	P—Rh—Cl	86.50 (4)
Cl—Rh—P—C31	68.31 (7)	Rh—P—C31—C32	−36.46 (16)

Symmetry code: (i) $-x, -y, -z$.

Table 2
Comparative X-ray crystallographic data for [RhCl(P*X*Ph$_2$)$_2$(CO)] complexes.

X	Rh—Cl	Rh—CO	Rh—*P*	Disorder
Ph[a]	2.380 (2)	1.759 (7)	2.328 (1)	yes
2-MePh[b]	2.381 (1)	1.808 (6)	2.328 (1)	no
2-MeOPh[c]	2.387 (1)	1.807 (2)	2.195 (1)	no
2-MeBenzoPh[d]	2.408 (1)	1.869 (1)	2.327 (5)	no
Tri-4-tolyl[e]	2.358 (1)	1.798 (5)	2.334 (1)	no
4-MePh[f]	2.397 (2)	1.795 (5)	2.332 (1)	yes

Notes: (*a*) Chen *et al.* (1991); (*b*) Suomalainen *et al.* (2001); (*c*) Suomalainen *et al.* (2000); (*d*) Dutta *et al.* (2003); (*e*) Otto *et al.* (1999); (*f*) title compound (I).

All H atoms were positioned geometrically (C—H = 0.93–0.96 Å) and allowed to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(parent). The disordered H atoms of the methyl unit were constrained to have occupancies of 0.5 at each of the two positions.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT-Plus* (Bruker, 2004); data reduction: *SAINT-Plus* and *XPREP* (Bruker 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2004); software used to prepare material for publication: *SHELXL97*.

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