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trans-Carbonylchlorobis[(2-naphthyloxy)-diphenylphosphine-*κP*]rhodium(**I**)

Leo Kirsten, Elizabeth Steyl, Jan H. Van der Westhuizen and Gideon Steyl*

Department of Chemistry, University of the Free State, PO Box 339, Bloemfontein 9300, South Africa

Correspondence e-mail: geds12@yahoo.com

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Key indicators

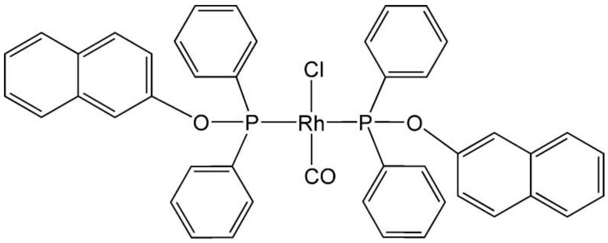
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.025
wR factor = 0.060
Data-to-parameter ratio = 15.9

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

The title compound, $[RhCl(C_{22}H_{34}OP)_2(CO)]$, can be described as one of a few Rh^I Vaska-type complexes containing a (2-naphthyloxy)diphenylphosphine ligand and has a square-planar geometry about the rhodium(I) metal centre, which lies on an inversion centre, so that the carbonyl and chloro ligands are disordered. The most important bond lengths and angle include Rh—P = 2.305 (1) Å, Rh—Cl = (*trans* CO) = 2.374 (2) Å, Rh—C (carbonyl) = 1.818 (6) Å and Rh—C—O = 178.1 (10)°. A weak π–π intermolecular stacking is observed, with an interplanar distance of 3.377 (2) Å.

Comment

A small number of crystal structures have been reported for monomolecular square-planar Rh^I complexes containing a chloro and phosphinite ligand ($POR Ph_2$, R any alkyl or aryl substituent) (Chahen *et al.*, 2005; Haar *et al.*, 1998). The title compound, (I), is presented as another example of an Rh^I Vaska-type complex containing a phosphinite ligand.



(I)

Compound (I) crystallizes with a square-planar geometry about the Rh^I metal centre (Fig. 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. The naphthyl substituent is directed back towards the Rh metal centre as indicated by the Cl—Rh—P—O1 and Rh—P—O1—C11 torsion angles (Table 1). Additional geometrical parameters that give an indication of the steric demand of the phosphinite ligand are the Tolman and effective solid-state cone angles (Otto *et al.*, 2000), which were calculated to be 162 and 161°, respectively.

Compound (I) is the only Rh^I Vaska-type complex of a phosphinite ligand to date that possesses the disordered packing mode of the carbonyl and chloro ligands (Table 2). The Rh—P, Rh—C and Rh—Cl bond distances are comparable with those in reported phosphinite complexes (Table 2). A weak π–π stacking between parallel naphthyl ring systems is observed, with an interplanar distance of 3.377 (2) Å (Fig. 2). This may be attributed to the extended planarity and the conformational flexibility of the naphthyl ring system. Additional C–H$\cdots\pi$ interactions are also observed between C110/

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H110 and ring C21–C26$(1 - x, 2 - y, 1 - z)$, and between C14/H14 and ring C31—C36$(1 + x, y, z)$, with H$\cdots$centroid distances of 2.826 (1) and 2.809 (1) Å, respectively.

Experimental

The title complex was synthesized by the addition of (2-NaphO)Ph_2P (28 mg, 0.085 mmol) to a 3 ml acetone solution of $[Rh(\mu\text{-}Cl)(CO)_2]_2$ (7.1 mg, 0.018 mmol). Slow evaporation of the solvent yielded yellow crystals of the desired product suitable for X-ray diffraction. Yield 24 mg (81%). IR spectrum (KBr) $\nu(CO) = 1974\ cm^{-1}$, (CH_2Cl_2) $\nu(CO) = 1991\ cm^{-1}$

Crystal data

$[RhCl(C_{22}H_{34}OP)_2(CO)]$
$M_r = 823.02$
Triclinic, $P\overline{1}$
$a = 9.4304$ (5) Å
$b = 10.2266$ (6) Å
$c = 11.0438$ (10) Å
$\alpha = 109.246$ (2)°
$\beta = 107.972$ (2)°
$\gamma = 99.024$ (2)°

$V = 915.99$ (11) Å^3
$Z = 1$
$D_x = 1.492\ Mg\ m^{-3}$
Mo $K\alpha$ radiation
$\mu = 0.67\ mm^{-1}$
$T = 100$ (2) K
Plate, yellow
$0.26 \times 0.12 \times 0.02$ mm

Data collection

Bruker X8 APEXII diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{min} = 0.845$, $T_{max} = 0.986$

11793 measured reflections
3982 independent reflections
3695 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 27.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.060$
$S = 1.07$
3982 reflections
250 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0215P)^2 + 0.66P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.50\ e\ Å^{-3}$
$\Delta\rho_{min} = -0.25\ e\ Å^{-3}$

Table 1
Selected geometric parameters (Å, °).

Rh—C01	1.818 (6)	P—C31	1.8102 (18)
Rh—P	2.3048 (5)	P—C21	1.8165 (19)
Rh—Cl	2.3743 (16)	O01—C01	1.080 (6)
P—O1	1.6314 (13)		
C01—Rh—P^i	92.0 (3)	O01—C01—Rh	178.1 (10)
P—Rh—Cl^i	93.16 (5)		
Cl—Rh—P—O1	−58.67 (7)	Rh—P—O1—C11	−27.55 (15)

Symmetry code: (i) $-x + 1, -y + 1, -z + 1$.

Table 2
Comparative X-ray crystallographic data for $[Rh(Cl)(CO)(POR Ph_2)_2]$ complexes.

R	Rh—Cl	Rh—CO	Rh—P	Disorder	Ref.
2,4,6-$Me_3C_6H_2$	2.341 (1)	1.834 (4)	2.320 (1)	no	*a*
$(CH_2)_2(CF_2)_5CF_3$	2.356 (1)	1.829 (5)	2.304 (1)	no	*b*
2-Naphthyl	2.374 (2)	1.818 (6)	2.305 (1)	yes	*c*

Notes: (*a*) Chahen *et al.* (2005); (*b*) Haar *et al.* (1998); (*c*) this work.

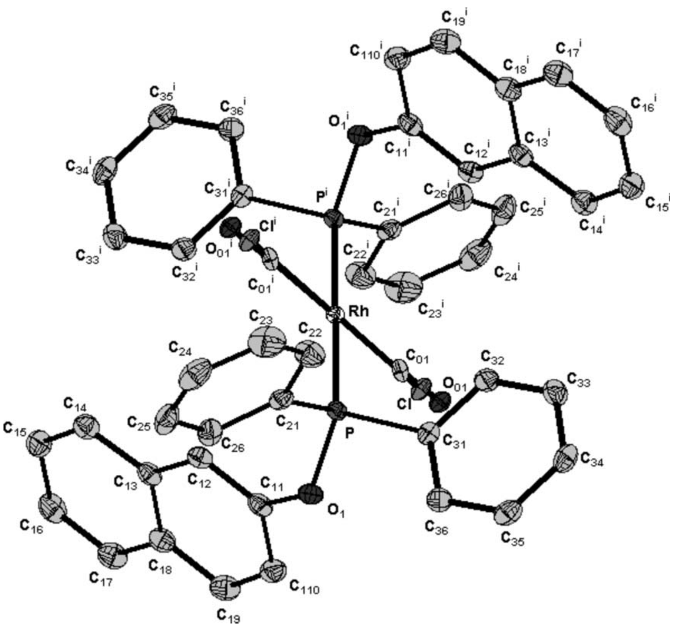

Figure 1
The molecular structure of (I), showing the disorder of the carbonyl group and chloro atom. For the carbon rings the first digit refers to ring number and the second digit to the atom in the ring. H atoms have been omitted for clarity [symmetry code: (i) $-x, -y, -z$].

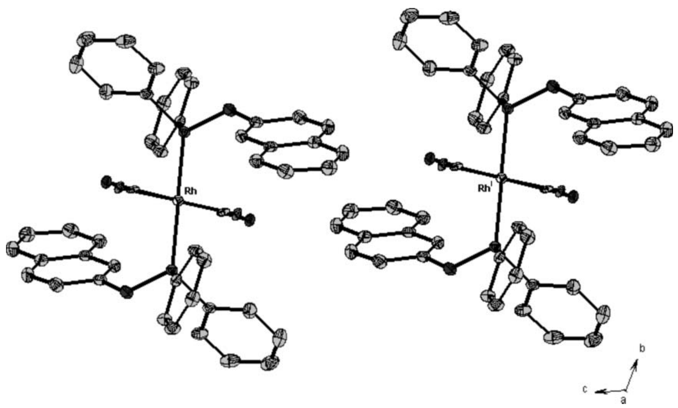

Figure 2
Part of the unit-cell contents, showing the π-stacking unit [symmetry code: (i) $x, y, -1 + z$].

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.95 Å and $U_{iso}(H) = 1.2U_{eq}$(parent).

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, 2006); software used to prepare material for publication: *SHELXL97*.

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