Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Carbonyl(cyclohexyldiphenylphosphine)-(8-hydroxyquinolinato)rhodium(I)

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Received 28 March 2006
Accepted 10 April 2006

The molecules of the title compound, [Rh(C_9H_6NO)($C_{18}H_{21}P$)(CO)], pack with the H atoms of the phenyl and cyclohexyl rings at distances of 2.80–2.85 Å from the adjacent oxine ligand plane. The effective cone angle (Θ_E) for the phosphine ligand is 151°. The bidentate oxine ligand has a bite angle of 79.41 (9)° and the Rh—P bond length is 2.2798 (8) Å.

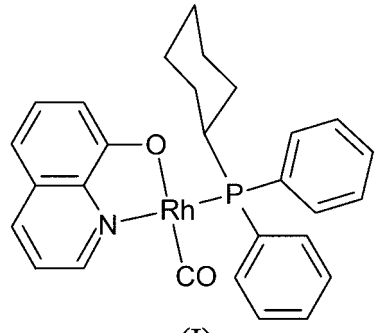
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.036
wR factor = 0.083
Data-to-parameter ratio = 20.0

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

Comment

In complexes of the type [Rh(OX)$(CO)_2$] (where OX = C_9H_6NO, 8-hydroxyquinolinate), mono-carbonyl substitution is achieved by addition of ligands such as (ER_3), where E = P, As or Sb, and R = alkyl, aryl, alkoyl or aroyl. In the bidentate ligand OX, the N and O donor atoms result in displacement of one carbonyl group and the most stable isomer having the ER_3 *trans* to the N atom (Janse van Rensburg *et al.*, 2005).



(I)

In the title compound, (I), the Rh atom is displaced by 0.062 (1) Å from the slightly distorted square-planar coordination polyhedron (r.m.s. displacement of fitted atoms = 0.098 Å; Fig. 1 and Table 1). A dihedral angle of 2.0 (1)° is observed between the benzene and pyridine rings of the oxine ligand, evidence of a slight distortion. A larger distortion from planarity is observed between the bidentate oxine backbone and the metal coordination plane, as evident from the dihedral angle of 11.3 (1)°. The C10—Rh—P and N—Rh—O1 angles are 88.70 (10) and 79.41 (9)°, respectively. This deviation from the ideal 90° illustrates the distorted square-planar coordination, which has also been observed in other oxinate complexes (Table 2).

Intramolecular C—H···O contacts (C11—H11···O1 = 109°, C12—H12*B*···O1 = 125°) are present, which might affect the orientation of the cyclohexyl ring. The molecules pack with the phenyl and cyclohexyl rings of the phosphine ligand directed towards the neighboring oxinate ligand (Fig. 2),

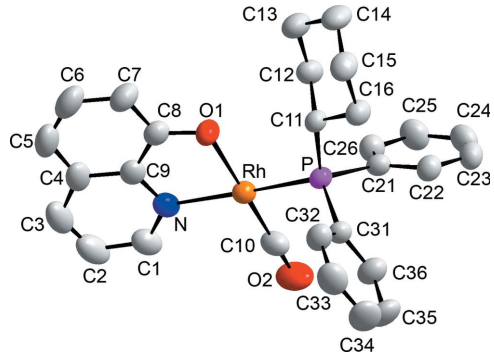


Figure 1
A view of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity.

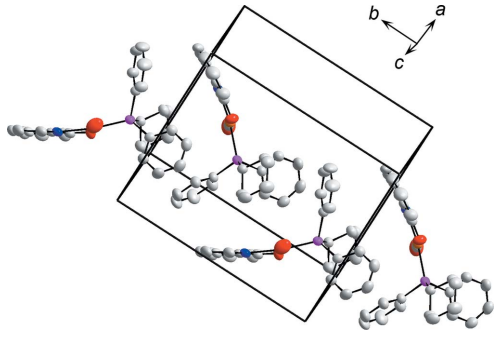


Figure 2
Unit-cell view, showing the phenyl and cyclohexyl rings directed towards the neighboring oxine ligand. H atoms have been omitted.

with the H atoms of the phenyl and cyclohexyl rings at a distance of 2.80–2.85 Å from the oxine ligand plane.

Using the Rh—P bond distance of 2.2798 (8) Å, the effective cone angle (Θ_E), as described previously (Tolman, 1977; Otto *et al.*, 2000), was calculated as 151°. In solution, the ligand substituent orientation might differ, as observed by Ferguson *et al.* (1978), resulting in a variation in cone angle size. Therefore, the solid-state cone angle might not necessarily be a true indication of the ligand steric properties in solution. The effective cone angle (Θ_E) of 151° is comparable with the average value of 153° for the corresponding Vaska-type palladium complex *trans*-$[PdCl_2(PPh_2Cy)_2]$ reported by Meij *et al.* (2003). Comparable $^1J_{(Rh-P)}$ values in (I) and *trans*-$[PdCl_2(PPh_2Cy)_2]$ illustrate that the presence of the cyclohexyl ring in (I) in place of one phenyl ring in *trans*-$[PdCl_2(PPh_2Cy)_2]$ has little or no electronic influence on the Rh—P bond.

Bond distances, bite angles and the effective cone angle for (I) are compared with those of similar structures in Table 2. A reasonable correlation between the bond distances and angles for the quinoline rings is observed. The expected differences in Rh—P bond lengths and coupling constants between phosphine and phosphite ligand complexes are observed, as reported by Janse van Rensburg *et al.* (2005). The increase in the Rh—P, Rh—N and Rh—O bond lengths of complex (I) when compared with complexes containing triphenylphosphine, emphasize the better σ-donor and weaker π-acceptor characteristics of the cyclohexyldiphenylphosphine ligand. The additional increase from entry 2 to 4 in Table 2 is due to the change from Rh^I to Rh^{III} following CH_3I oxidative addition.

Experimental

$[RhCl(CO)_2]_2$ was prepared according to a literature method (McCleverty & Wilkinson, 1990). Other chemicals and solvents were obtained from Sigma–Aldrich and used as received. $[Rh(OX)(CO)_2]$ was synthesized by mixing solutions of 8-hydroxyquinoline (30 mg, 0.206 mmol) in dimethylformamide (DMF, 1 ml) and $[RhCl(CO)_2]_2$ (37 mg, 0.094 mmol) in DMF (1 ml). Upon addition of ice–water (20 ml), the complex precipitated and was filtered off. Ligand substitution on the complex $[Rh(OX)(CO)_2]$ was performed by dissolving (30 mg, 0.056 mmol) in acetone (40 ml) followed by slow addition of $P(C_{18}H_{21})$ (18 mg, 0.067 mmol) in acetone (2 ml) (yield 30 mg, 69%). $^{31}P\{H\}$ NMR ($CDCl_3$, 121.5 MHz, p.p.m.): 47.7 [$^1J_{(Rh-P)}$ = 163.5 Hz]; IR (acetone) ν(CO): 1960 cm^{-1}; (KBr) ν(CO): 1968 cm^{-1}.

Crystal data

$[Rh(C_9H_6NO)(C_{18}H_{21}P)(CO)]$
M_r = 543.39
Monoclinic, $P2_1/n$
a = 10.559 (2) Å
b = 13.296 (3) Å
c = 17.658 (4) Å
β = 90.99 (3)°
V = 2478.7 (9) Å^3
Z = 4
D_x = 1.456 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.78 mm^{-1}
T = 293 (2) K
Cuboid, yellow
0.20 × 0.18 × 0.14 mm

Data collection

Bruker SMART 1K CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.860, T_{max} = 0.899
16521 measured reflections
5979 independent reflections
4036 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 28°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.083
S = 1.00
5979 reflections
299 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0365P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Rh—C10	1.803 (3)	P—C21	1.838 (3)
Rh—O1	2.060 (2)	P—C31	1.844 (3)
Rh—N	2.128 (2)	P—C11	1.846 (3)
Rh—P	2.2798 (8)	O2—C10	1.161 (4)
C10—Rh—N	99.10 (12)	C31—P—Rh	112.15 (9)
O1—Rh—N	79.41 (9)	C11—P—Rh	110.48 (9)
C10—Rh—P	88.70 (10)	C16—C11—P	118.41 (19)
O1—Rh—P	93.11 (6)	C12—C11—P	109.19 (19)
C21—P—Rh	118.42 (9)		

Table 2
Comparative geometric data (Å, °) for [Rh(O*X*)(PR_3)(CO)] complexes..

PR_3	Rh—P	Rh—N	Rh—O	N—Rh—O	Θ_E (Å)	$^1J_{Rh-P}$ (Hz)
PPh_2Cy[i]	2.279 (1)	2.128 (2)	2.060 (2)	79.41 (9)	151	163
PPh_3[ii]	2.261 (2)	2.098 (9)	2.042 (5)	80.0 (3)	153	161
$P(OPh)_3$[iii]	2.186 (1)	2.097 (2)	2.022 (2)	80.8[iv]	154	281
PPh_3[v]	2.317 (2)	2.084 (7)	2.037 (4)	81.2 (2)	153	163
$P(O2,6DMP)_3$[vi]	2.198 (1)	2.091 (3)	2.029 (3)	80.3 (1)	183	280

References and notes: (i) this work, Ph_2Cy = cyclohexyldiphenyl; (ii) Leipoldt *et al.* (1981); (iii) Simanko *et al.* (2000); (iv) data extracted from the Cambridge Structural Database (Version 5.26), no s.u. values (Allen, 2002); (v) van Aswegen *et al.* (1991); Rh^{III} iodomethane oxidative addition product containing apical *trans* methyl and iodo ligands; (vi) Janse van Rensburg *et al.* (2005).

H atoms were positioned geometrically and allowed to ride during subsequent refinement, with C—H = 0.93, 0.97 and 0.98 Å for those on phenyl, secondary and tertiary C atoms, respectively. In each case, $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART-NT* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus* and *XPREP* (Bruker, 1999); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg & Berndt, 2001); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Financial assistance from the South African National Research Foundation, the Research Fund of the University of the Free State and SASOL is gratefully acknowledged. The University of the Witwatersrand (Professor D. Levendis and Dr D. G. Billing) is thanked for the use of its diffractometer. Part of this material is based on work supported by the South African National Research Foundation [SA NRF, GUN 2038915] (University of the Free State). Opinions, findings, conclusions or recommendations expressed in this material are those of the authors and do not necessarily reflect the views of the NRF.

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