

Carbonyl(5-chloroquinolin-8-olato- κ^2N,O)[tris(4-chlorophenyl)phosphine- κP]rhodium(I)

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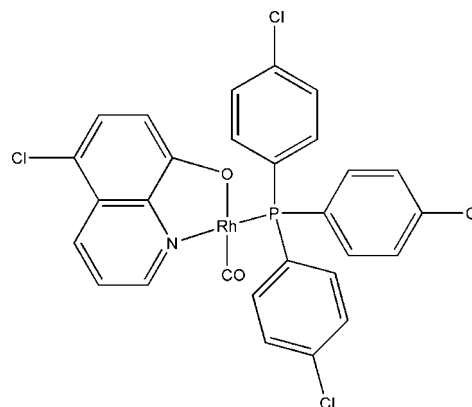
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(C-C) = 0.004$ Å; R factor = 0.036; wR factor = 0.068; data-to-parameter ratio = 20.1.

In the title compound, $[Rh(C_9H_7ClNO)(C_{18}H_{12}Cl_3P)(CO)]$, the phosphine ligand is *trans* to the N atom of the quinolinolate ligand. This indicates that changes in the electronic properties of the quinolinolate backbone have a negligible influence on the phosphine substitution. Important geometrical parameters are the quinolinolate bite angle of $80.15(9)^\circ$ and the Rh–P bond distance of $2.2478(9)$ Å, the effective cone angle (Θ_E) for the phosphine ligand being 165° . Quinoline ligand-to-ligand π -stacking is in a tail-to-tail fashion with an intermolecular distance of 3.26 Å. The molecule also exhibits intramolecular C–H \cdots Cl and C–H \cdots O hydrogen bonds.

Related literature

For the neutral 5-chloro-8-hydroxyquinoline ligand structure, see: Banerjee & Saha (1986). For an example of a β -diketonatorhodium(I) phosphine complex, see: Brink *et al.* (2007). For related quinolinatorhodium(I)phosphite complexes, see: Janse van Rensburg *et al.* (2005*a,b*, 2006*a*). For quinolinatorhodium(I) phosphine complexes, see Janse van Rensburg & Roodt (2006); Janse van Rensburg *et al.* (2006*b*). For related literature, see: Allen (2002); Domenicano *et al.* (1975); Hughes & Truter (1979); McCleverty & Wilkinson (1990); Otto *et al.* (2000); Tolman (1977).



Experimental

Crystal data

$[Rh(C_9H_7ClNO)(C_{18}H_{12}Cl_3P)(CO)]$
 $M_r = 675.11$
 Monoclinic, $P2_1/c$
 $a = 9.558(5)$ Å
 $b = 19.788(5)$ Å
 $c = 14.608(5)$ Å
 $\beta = 103.670(5)^\circ$

$V = 2684.6(18)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 1.12$ mm⁻¹
 $T = 100(2)$ K
 $0.12 \times 0.08 \times 0.06$ mm

Data collection

Bruker Kappa APEXII diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2004)
 $T_{min} = 0.877$, $T_{max} = 0.946$

39703 measured reflections
 6708 independent reflections
 4272 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.097$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.068$
 $S = 0.88$
 6708 reflections

334 parameters
 H-atom parameters constrained
 $\Delta\rho_{max} = 0.68$ e Å⁻³
 $\Delta\rho_{min} = -0.74$ e Å⁻³

Table 1

Selected bond lengths (Å).

C10–Rh	1.803(4)	O1–Rh	2.038(2)
N–Rh	2.093(2)	P–Rh	2.2478(9)

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C3–H3 \cdots Cl	0.93	2.77	3.132(4)	104
C22–H22 \cdots O1	0.93	2.27	3.111(4)	149

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: SIR97 (Altomare *et al.*, 1999); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: DIAMOND (Brandenburg & Putz, 2005); software used to prepare material for publication: WinGX (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2393).

References

- Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.
- Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
- Banerjee, T. & Saha, N. N. (1986). *Acta Cryst.* **C42**, 1408–1411.
- Brandenburg, K. & Putz, H. (2005). *DIAMOND*. Release 3.1b. Crystal Impact GbR, Bonn, Germany.
- Brink, A., Roodt, A. & Visser, H. G. (2007). *Acta Cryst.* **E63**, m48–m50.
- Bruker (2004). *SADABS* (Version 2004/1) and *SAINT-Plus* (Version 7.12 including *XPREP*). Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2005). *APEX2*. Version 1.0-27. Bruker AXS Inc., Madison, Wisconsin, USA.
- Domenicano, A., Vaciago, A. & Coulson, C. A. (1975). *Acta Cryst.* **B31**, 1630–1641.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Hughes, D. L. & Truter, M. R. (1979). *J. Chem. Soc. Dalton Trans.* pp. 520–527.
- Janse van Rensburg, J. M. & Roodt, A. (2006). *Acta Cryst.* **E62**, m2981–m2983.
- Janse van Rensburg, J. M., Roodt, A. & Muller, A. (2006a). *Acta Cryst.* **E62**, m2978–m2980.
- Janse van Rensburg, J. M., Roodt, A. & Muller, A. (2006b). *Acta Cryst.* **E62**, m1040–m1042.
- Janse van Rensburg, J. M., Roodt, A., Muller, A. & Meijboom, R. (2005a). *Acta Cryst.* **E61**, m1741–m1743.
- Janse van Rensburg, J. M., Roodt, A., Muller, A. & Meijboom, R. (2005b). *Acta Cryst.* **E61**, m2743–m2745.
- McCleverty, J. A. & Wilkinson, G. (1990). *Inorg. Synth.* **28**, 84–86.
- Otto, S., Roodt, A. & Smith, J. (2000). *Inorg. Chim. Acta*, **303**, 295–299.
- Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
- Tolman, C. A. (1977). *Chem. Rev.* **77**, 313–348.

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Carbonyl(5-chloroquinolin-8-olato- κ^2N,O)[tris(4-chlorophenyl)phosphine- κP]rhodium(I)

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Comment

Bonding of bidentate ligands to transition metals is a well known concept. One of the most common bidentate examples are probably the β -diketones, *e.g.* acetylacetonone, with 42 entries in the CSD for Rh(I) complexes alone [Cambridge Structural Database, Version 5.28, May 2007 (Allen, 2002)]. Bidentate ligands with different donor atoms such as 8-hydroxyquinoline and its derivatives also form chelating ring systems with transition metals, *via* the N, O donor atoms. To date there are six 8-hydroxyquinolinatorrhodium(I) complexes deposited in the CSD.

In the title compound [Rh(C₉H₇ClNO)(CO){P(C₆H₄Cl)₃}], the fused ring system of the bidentate ligand is almost planar, with an r.m.s. deviation from planarity of 0.0423 Å and a dihedral angle of 4.87 (16) Å between the benzene and pyridine rings. The 5'-chloro substituent, the O-donor atom and the metal centre are essentially in the plane of the quinoline system, displaced by 0.124 (3) Å, 0.081 (3) Å and 0.065 (3) Å respectively. The rhodium metal centre is slightly displaced from the coordination plane by 0.014 (1) Å (r.m.s. of fitted atoms = 0.0007 Å). The N...O bite distance is 2.659 (9) Å and the N—Rh—O bite angle is 80.15 (9)°. The Rh—P bond distance is 2.2478 (9) Å, while the C8—O1 bond distance is 1.326 (3) Å and the endocyclic angle at C7—C8—C9 = 117.8 (3)°. These are comparable to the average distances for rhodium(I)quinolinato complexes reported previously (Janse van Rensburg *et al.*, 2005*a,b*, 2006, *a,b*, Janse van Rensburg & Roodt, 2006).

The metal-carbonyl is slightly bent with a Rh—C10—O2 bond angle of 177.4 (3)°, and a C10—O2 bond distance of 1.154 (4) Å, also comparable with previous reported rhodium(I)quinolinol complexes. Phosphine ligand substitution occurred *trans* to the N-donor atom which is the stronger σ -donor. Phosphine substituent arrangement is in such a way that one of the phenyl rings is *cis* to the carbonyl, reflected by the C10—Rh—P—C11 torsion angle of 1.86 (14)°. The steric behavior of the ligand at the metal centre was determined by calculating the effective cone angle as described previously (Tolman, 1977; Otto *et al.*, 2000). A value of 165° was obtained.

Short intramolecular contacts are present between C3...Cl with C3—H...Cl = 104° and C3...Cl = 3.132 (4) Å and between C22...O1 with C22—H...O1 = 149.3° and C22...O1 = 3.111 (4) Å. The familiar quinoline ligand to ligand stacking fashion for these type of compounds is also present, stabilized with a π -stacking distance of 3.26 Å between the planes defined by the C1...C9, N atoms of adjacent quinoline ligands; in addition, there are two Rh...H contacts (3.388 Å) and two Cl...O contacts (3.489 Å).

Banerjee and Saha reported the effects on bond lengths and angles of the free 5'-chloro moiety *versus* that of the free 8-hydroxyquinoline ligand (Banerjee & Saha, 1986). In this article we compare these to the rhodium bonded 5-chloro-8-hydroxyquinoline title compound (Table 1). There is a *ca* 0.1 Å decrease in the N...O bite distance of the title compound, 2.659 (9) Å, when compared to that of the free ligand. Chelation of 5-chloro-8-hydroxyquinoline to the metal centre shortens the C8—O1 bond distance by *ca* 0.04 Å, compared to the neutral 8-hydroxyquinolines (Hughes & Truter, 1979).

supplementary materials

A decrease is also noted in the C6—C7—C8 ring angle when comparing the title compound with the free 5-chloro-8-hydroxyquinoline and unsubstituted 8-hydroxyquinoline, (117.8 (3)°, 119.6 (5)° and 121.4 (4)°), respectively. Thus the smaller the endocyclic ring angle, the stronger conjugation is observed between the benzene and the oxygen (O1) (Banerjee & Saha, 1986; Domenicano *et al.*, 1975).

Experimental

Chemicals and solvents were obtained from Sigma-Aldrich and used as received. $[\text{RhCl}(\text{CO})_2]_2$ was prepared according to the literature method (McCleverty & Wilkinson, 1990). $[\text{Rh}(\text{5ClOX})(\text{CO})_2]$ was synthesized by mixing solutions of 5-chloro-8-hydroxyquinoline, (5ClOX), (61.4 mg, 0.344 mmol) in DMF (1 ml) and $[\text{RhCl}(\text{CO})_2]_2$ (60.9 mg, 0.156 mmol) in DMF (1 ml). Upon addition of ice water (30 ml) the product precipitated and was filtered. Ligand substitution on the complex $[\text{Rh}(\text{5ClOX})(\text{CO})_2]$ was performed by dissolving (50 mg, 0.148 mmol) in acetone (40 ml) followed by slow addition of $\text{P}(\text{4-ClC}_6\text{H}_4)_3$ (55 mg, 0.163 mmol) in acetone (10 ml). Upon evaporation, crystals suitable for single-crystal X-ray crystallography were obtained (yield: 72 mg, 76%).

Spectroscopic data: $^{31}\text{P}\{\text{H}\}$ NMR (CDCl_3 , 121.447 MHz, p.p.m.): 40.47 p.p.m. [$^1J_{(\text{Rh}-\text{P})} = 166$ Hz]; IR (KBr) $\nu(\text{CO})$: 1965 cm^{-1} .

Refinement

The H atoms were positioned geometrically and refined using a riding model with fixed C—H distances of 0.93 Å (CH) [$U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}$].

The highest density peak is 0.68 located 0.95 Å from C10 and the deepest hole is -0.74 located at 0.56 Å from Rh.

Figures

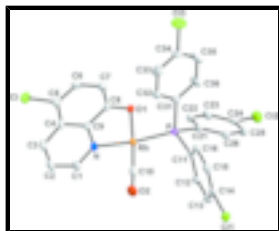


Fig. 1. A view of (I) showing the atom-numbering scheme with displacement ellipsoids at the 30% probability level. H atoms have been omitted for clarity.

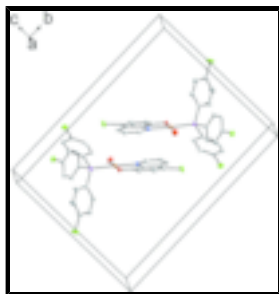


Fig. 2. Part of the unit-cell contents viewed along the *a* axis, showing the quinoline ligand to ligand π -stacking.

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Crystal data

[Rh(C ₉ H ₇ ClNO)(C ₁₈ H ₁₂ Cl ₃ P)(CO)]	$F_{000} = 1344$
$M_r = 675.11$	$D_x = 1.67 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
Hall symbol: -P 2ybc	$\lambda = 0.71073 \text{ \AA}$
$a = 9.558 (5) \text{ \AA}$	Cell parameters from 5536 reflections
$b = 19.788 (5) \text{ \AA}$	$\theta = 2.5\text{--}28.1^\circ$
$c = 14.608 (5) \text{ \AA}$	$\mu = 1.12 \text{ mm}^{-1}$
$\beta = 103.670 (5)^\circ$	$T = 100 (2) \text{ K}$
$V = 2684.6 (18) \text{ \AA}^3$	Cuboid, yellow
$Z = 4$	$0.12 \times 0.08 \times 0.06 \text{ mm}$

Data collection

Bruker X8 APEXII 4K Kappa CCD diffractometer	4272 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.097$
$T = 100(2) \text{ K}$	$\theta_{\text{max}} = 28.4^\circ$
ω & φ scans	$\theta_{\text{min}} = 1.8^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2004)	$h = -12 \rightarrow 12$
$T_{\text{min}} = 0.877$, $T_{\text{max}} = 0.946$	$k = -26 \rightarrow 26$
39703 measured reflections	$l = -19 \rightarrow 19$
6708 independent reflections	

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0221P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.036$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.068$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 0.88$	$\Delta\rho_{\text{max}} = 0.68 \text{ e \AA}^{-3}$
6708 reflections	$\Delta\rho_{\text{min}} = -0.74 \text{ e \AA}^{-3}$
334 parameters	Extinction correction: none

Special details

Experimental. The intensity data was collected on a Bruker X8 Apex II 4 K Kappa CCD diffractometer using an exposure time of 40 s/frame. A total of 1348 frames were collected with a frame width of 0.5° covering up to $\theta = 28.36^\circ$ with 99.7% completeness accomplished.

supplementary materials

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	-0.1624 (4)	0.04276 (15)	-0.0917 (2)	0.0211 (7)
H1	-0.2456	0.052	-0.0713	0.025*
C2	-0.1670 (4)	-0.00805 (15)	-0.1592 (2)	0.0248 (8)
H2	-0.2516	-0.0319	-0.1827	0.03*
C3	-0.0468 (4)	-0.02217 (15)	-0.1899 (2)	0.0240 (8)
H3	-0.0498	-0.0551	-0.2358	0.029*
C4	0.0823 (4)	0.01282 (14)	-0.15262 (19)	0.0206 (7)
C5	0.2164 (4)	0.00051 (15)	-0.1737 (2)	0.0260 (8)
C6	0.3369 (4)	0.03361 (16)	-0.1267 (2)	0.0291 (8)
H6	0.4249	0.023	-0.1398	0.035*
C7	0.3318 (4)	0.08330 (16)	-0.0591 (2)	0.0262 (8)
H7	0.4162	0.1048	-0.0278	0.031*
C8	0.2036 (4)	0.10045 (14)	-0.03872 (19)	0.0189 (7)
C9	0.0780 (4)	0.06354 (15)	-0.08460 (19)	0.0183 (7)
C10	-0.1950 (4)	0.16063 (16)	0.05666 (19)	0.0214 (7)
C11	-0.0802 (3)	0.27579 (14)	0.20064 (19)	0.0143 (6)
C12	-0.1370 (3)	0.23426 (15)	0.2598 (2)	0.0188 (7)
H12	-0.0998	0.191	0.2733	0.023*
C13	-0.2469 (3)	0.25576 (16)	0.2985 (2)	0.0215 (7)
H13	-0.2825	0.228	0.3391	0.026*
C14	-0.3032 (3)	0.31923 (16)	0.27585 (19)	0.0194 (7)
C15	-0.2514 (3)	0.36159 (15)	0.21715 (19)	0.0205 (7)
H15	-0.2921	0.404	0.2021	0.025*
C16	-0.1378 (3)	0.34030 (16)	0.18070 (18)	0.0189 (7)
H16	-0.0996	0.3692	0.1427	0.023*
C21	0.2029 (3)	0.22046 (14)	0.24806 (19)	0.0146 (6)
C22	0.3095 (3)	0.17624 (14)	0.2356 (2)	0.0189 (7)
H22	0.3046	0.1575	0.1765	0.023*
C23	0.4229 (3)	0.15985 (16)	0.3101 (2)	0.0235 (7)
H23	0.4941	0.1301	0.3016	0.028*
C24	0.4294 (3)	0.18803 (15)	0.3974 (2)	0.0197 (7)
C25	0.3261 (3)	0.23252 (15)	0.4116 (2)	0.0202 (7)
H25	0.3321	0.2514	0.4707	0.024*
C26	0.2132 (3)	0.24863 (15)	0.3365 (2)	0.0183 (7)
H26	0.143	0.2788	0.3454	0.022*
C31	0.1206 (3)	0.31427 (14)	0.09282 (19)	0.0170 (7)
C32	0.0440 (4)	0.33531 (16)	0.0045 (2)	0.0246 (7)
H32	-0.0355	0.3106	-0.0273	0.029*
C33	0.0851 (4)	0.39297 (17)	-0.0367 (2)	0.0340 (9)
H33	0.0324	0.4077	-0.0952	0.041*

C34	0.2040 (4)	0.42767 (16)	0.0099 (2)	0.0267 (8)
C35	0.2825 (4)	0.40741 (17)	0.0963 (2)	0.0359 (9)
H35	0.3643	0.4312	0.1267	0.043*
C36	0.2384 (4)	0.35094 (17)	0.1379 (2)	0.0313 (8)
H36	0.2896	0.3376	0.1975	0.038*
N	-0.0451 (3)	0.07802 (12)	-0.05580 (16)	0.0179 (6)
O1	0.1899 (2)	0.14853 (10)	0.02175 (13)	0.0211 (5)
O2	-0.3131 (2)	0.16127 (12)	0.06297 (15)	0.0329 (6)
P	0.05603 (9)	0.24118 (4)	0.14644 (5)	0.01512 (18)
Cl1	-0.44424 (8)	0.34634 (4)	0.32268 (5)	0.02688 (18)
Cl	0.22850 (11)	-0.05749 (4)	-0.26151 (6)	0.0387 (2)
Cl2	0.57460 (9)	0.16895 (4)	0.49090 (6)	0.0322 (2)
Cl3	0.25882 (13)	0.49923 (5)	-0.04213 (7)	0.0507 (3)
Rh	-0.01248 (3)	0.156302 (12)	0.043330 (15)	0.01617 (7)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0222 (19)	0.0186 (17)	0.0196 (16)	-0.0004 (15)	-0.0007 (14)	0.0037 (13)
C2	0.032 (2)	0.0150 (16)	0.0213 (16)	-0.0053 (16)	-0.0052 (15)	0.0011 (13)
C3	0.042 (2)	0.0131 (16)	0.0145 (15)	-0.0019 (16)	0.0023 (15)	-0.0019 (12)
C4	0.038 (2)	0.0090 (15)	0.0145 (15)	-0.0037 (15)	0.0057 (14)	0.0019 (12)
C5	0.045 (2)	0.0142 (16)	0.0249 (17)	-0.0032 (16)	0.0201 (16)	-0.0040 (13)
C6	0.038 (2)	0.0186 (17)	0.039 (2)	-0.0054 (17)	0.0253 (18)	-0.0039 (15)
C7	0.030 (2)	0.0229 (18)	0.0283 (18)	-0.0086 (16)	0.0128 (16)	-0.0053 (14)
C8	0.031 (2)	0.0121 (15)	0.0158 (15)	-0.0055 (14)	0.0090 (14)	0.0006 (12)
C9	0.029 (2)	0.0122 (15)	0.0140 (14)	-0.0037 (14)	0.0066 (14)	0.0027 (12)
C10	0.030 (2)	0.0153 (16)	0.0162 (15)	0.0012 (17)	-0.0008 (14)	-0.0030 (13)
C11	0.0139 (16)	0.0131 (15)	0.0139 (14)	-0.0017 (13)	-0.0006 (12)	-0.0025 (11)
C12	0.0174 (18)	0.0145 (16)	0.0241 (16)	0.0012 (14)	0.0038 (14)	0.0029 (13)
C13	0.0219 (19)	0.0223 (17)	0.0203 (16)	-0.0057 (15)	0.0050 (14)	0.0018 (13)
C14	0.0148 (17)	0.0262 (17)	0.0162 (15)	-0.0019 (14)	0.0017 (13)	-0.0061 (13)
C15	0.0260 (19)	0.0152 (16)	0.0189 (16)	0.0042 (14)	0.0026 (14)	-0.0022 (12)
C16	0.0247 (17)	0.0170 (15)	0.0144 (14)	-0.0057 (16)	0.0033 (12)	-0.0019 (13)
C21	0.0162 (17)	0.0114 (15)	0.0154 (15)	-0.0008 (13)	0.0019 (12)	0.0023 (11)
C22	0.0228 (18)	0.0154 (16)	0.0188 (15)	0.0000 (14)	0.0058 (13)	-0.0034 (12)
C23	0.0232 (18)	0.0142 (15)	0.0317 (17)	0.0053 (16)	0.0038 (14)	-0.0033 (14)
C24	0.0173 (18)	0.0180 (16)	0.0222 (16)	-0.0057 (14)	0.0015 (14)	0.0076 (13)
C25	0.0227 (19)	0.0233 (18)	0.0143 (15)	0.0000 (15)	0.0040 (13)	0.0000 (13)
C26	0.0173 (17)	0.0158 (16)	0.0228 (16)	0.0025 (14)	0.0064 (13)	0.0002 (13)
C31	0.0221 (18)	0.0128 (15)	0.0183 (15)	0.0008 (14)	0.0091 (13)	-0.0004 (12)
C32	0.0297 (19)	0.0237 (18)	0.0196 (15)	-0.0088 (16)	0.0046 (14)	-0.0001 (14)
C33	0.046 (3)	0.034 (2)	0.0207 (18)	-0.0048 (19)	0.0046 (17)	0.0100 (15)
C34	0.036 (2)	0.0156 (17)	0.0323 (19)	-0.0041 (16)	0.0157 (17)	0.0097 (14)
C35	0.033 (2)	0.028 (2)	0.041 (2)	-0.0162 (18)	-0.0041 (17)	0.0070 (16)
C36	0.032 (2)	0.0269 (19)	0.0283 (17)	-0.0092 (18)	-0.0065 (15)	0.0109 (15)
N	0.0257 (16)	0.0123 (13)	0.0137 (12)	-0.0017 (12)	0.0003 (11)	0.0014 (10)
O1	0.0287 (13)	0.0174 (11)	0.0198 (10)	-0.0066 (11)	0.0111 (9)	-0.0057 (9)

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O2	0.0242 (14)	0.0392 (15)	0.0337 (13)	-0.0024 (13)	0.0037 (11)	-0.0060 (12)
P	0.0190 (4)	0.0113 (4)	0.0148 (4)	-0.0008 (3)	0.0034 (3)	-0.0005 (3)
Cl1	0.0214 (4)	0.0336 (5)	0.0269 (4)	0.0015 (4)	0.0080 (3)	-0.0069 (4)
Cl	0.0581 (7)	0.0247 (5)	0.0409 (5)	-0.0090 (5)	0.0271 (5)	-0.0160 (4)
Cl2	0.0282 (5)	0.0343 (5)	0.0284 (4)	0.0036 (4)	-0.0046 (4)	0.0078 (4)
Cl3	0.0676 (8)	0.0303 (5)	0.0560 (6)	-0.0130 (5)	0.0181 (6)	0.0206 (5)
Rh	0.02153 (13)	0.01224 (11)	0.01413 (11)	-0.00259 (12)	0.00298 (9)	-0.00139 (10)

Geometric parameters (Å, °)

C1—N	1.319 (4)	C15—H15	0.93
C1—C2	1.401 (4)	C16—H16	0.93
C1—H1	0.93	C21—C22	1.387 (4)
C2—C3	1.357 (5)	C21—C26	1.389 (4)
C2—H2	0.93	C21—P	1.833 (3)
C3—C4	1.408 (4)	C22—C23	1.381 (4)
C3—H3	0.93	C22—H22	0.93
C4—C5	1.409 (5)	C23—C24	1.379 (4)
C4—C9	1.420 (4)	C23—H23	0.93
C5—C6	1.361 (5)	C24—C25	1.375 (4)
C5—Cl	1.745 (3)	C24—Cl2	1.744 (3)
C6—C7	1.403 (4)	C25—C26	1.382 (4)
C6—H6	0.93	C25—H25	0.93
C7—C8	1.370 (5)	C26—H26	0.93
C7—H7	0.93	C31—C36	1.371 (4)
C8—O1	1.326 (3)	C31—C32	1.388 (4)
C8—C9	1.430 (4)	C31—P	1.820 (3)
C9—N	1.370 (4)	C32—C33	1.390 (4)
C10—O2	1.154 (4)	C32—H32	0.93
C10—Rh	1.803 (4)	C33—C34	1.364 (5)
C11—C12	1.392 (4)	C33—H33	0.93
C11—C16	1.393 (4)	C34—C35	1.368 (4)
C11—P	1.811 (3)	C34—Cl3	1.746 (3)
C12—C13	1.372 (4)	C35—C36	1.384 (4)
C12—H12	0.93	C35—H35	0.93
C13—C14	1.376 (4)	C36—H36	0.93
C13—H13	0.93	N—Rh	2.093 (2)
C14—C15	1.373 (4)	O1—Rh	2.038 (2)
C14—Cl1	1.734 (3)	P—Rh	2.2478 (9)
C15—C16	1.383 (4)		
N—C1—C2	122.8 (3)	C23—C22—C21	120.5 (3)
N—C1—H1	118.6	C23—C22—H22	119.7
C2—C1—H1	118.6	C21—C22—H22	119.7
C3—C2—C1	119.5 (3)	C24—C23—C22	119.3 (3)
C3—C2—H2	120.2	C24—C23—H23	120.4
C1—C2—H2	120.2	C22—C23—H23	120.4
C2—C3—C4	120.2 (3)	C25—C24—C23	121.4 (3)
C2—C3—H3	119.9	C25—C24—Cl2	119.2 (2)
C4—C3—H3	119.9	C23—C24—Cl2	119.4 (2)

C3—C4—C5	126.2 (3)	C24—C25—C26	118.8 (3)
C3—C4—C9	116.8 (3)	C24—C25—H25	120.6
C5—C4—C9	117.0 (3)	C26—C25—H25	120.6
C6—C5—C4	120.8 (3)	C25—C26—C21	121.1 (3)
C6—C5—C1	119.5 (3)	C25—C26—H26	119.5
C4—C5—C1	119.8 (3)	C21—C26—H26	119.5
C5—C6—C7	121.8 (3)	C36—C31—C32	118.7 (3)
C5—C6—H6	119.1	C36—C31—P	122.5 (2)
C7—C6—H6	119.1	C32—C31—P	118.7 (2)
C8—C7—C6	120.5 (3)	C31—C32—C33	120.6 (3)
C8—C7—H7	119.7	C31—C32—H32	119.7
C6—C7—H7	119.7	C33—C32—H32	119.7
O1—C8—C7	123.8 (3)	C34—C33—C32	119.0 (3)
O1—C8—C9	118.4 (3)	C34—C33—H33	120.5
C7—C8—C9	117.8 (3)	C32—C33—H33	120.5
N—C9—C4	122.1 (3)	C33—C34—C35	121.6 (3)
N—C9—C8	115.9 (3)	C33—C34—Cl3	119.4 (3)
C4—C9—C8	121.9 (3)	C35—C34—Cl3	118.9 (3)
O2—C10—Rh	177.4 (3)	C34—C35—C36	118.9 (3)
C12—C11—C16	118.5 (3)	C34—C35—H35	120.5
C12—C11—P	118.3 (2)	C36—C35—H35	120.5
C16—C11—P	123.0 (2)	C31—C36—C35	121.2 (3)
C13—C12—C11	121.4 (3)	C31—C36—H36	119.4
C13—C12—H12	119.3	C35—C36—H36	119.4
C11—C12—H12	119.3	C1—N—C9	118.6 (3)
C12—C13—C14	118.6 (3)	C1—N—Rh	129.8 (2)
C12—C13—H13	120.7	C9—N—Rh	111.65 (19)
C14—C13—H13	120.7	C8—O1—Rh	113.82 (19)
C15—C14—C13	121.9 (3)	C11—P—C31	103.86 (14)
C15—C14—Cl1	119.1 (2)	C11—P—C21	102.88 (13)
C13—C14—Cl1	119.0 (2)	C31—P—C21	104.69 (14)
C14—C15—C16	119.1 (3)	C11—P—Rh	116.73 (10)
C14—C15—H15	120.4	C31—P—Rh	112.33 (10)
C16—C15—H15	120.4	C21—P—Rh	114.94 (10)
C15—C16—C11	120.4 (3)	C10—Rh—O1	176.92 (11)
C15—C16—H16	119.8	C10—Rh—N	96.90 (12)
C11—C16—H16	119.8	O1—Rh—N	80.15 (9)
C22—C21—C26	118.9 (3)	C10—Rh—P	91.62 (10)
C22—C21—P	118.7 (2)	O1—Rh—P	91.33 (6)
C26—C21—P	122.4 (2)	N—Rh—P	171.45 (8)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C3—H3...Cl	0.93	2.77	3.132 (4)	104
C22—H22...O1	0.93	2.27	3.111 (4)	149

Table 3

supplementary materials

Comparative geometrical data (Å, °) for selected quinoline ligands.

System	N...O	C—O(hydroxyl)	Endocyclic ring angle (°) C7—C8—C9
Rh(OXCl)COP(4-ClPh) ₃ (i)	2.659 (9)	1.326 (3)	117.8 (3)
OXCl(ii)	2.747 (7)	1.346 (7)	119.6 (5)
OX(ii)	2.742 (4)	1.367 (5)	121.4 (4)

(i) This work; (OXCl) = 5-chloro-8-hydroxyquinoline and (4-ClC₆H₄) = *p*-chlorophenyl; (ii) Banerjee & Saha (1986); (OX) = 8-hydroxyquinoline.

Fig. 1

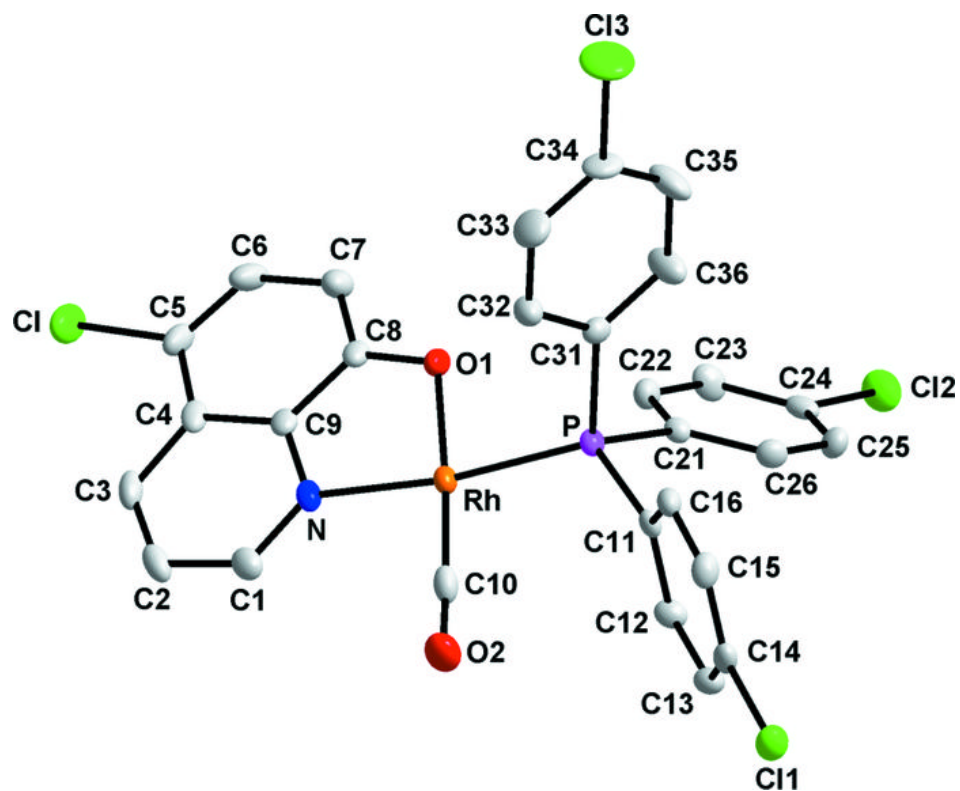


Fig. 2

