Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(Acetylacetonato-κ^2O,O')carbonyl-[2-(diphenylphosphino)pyridine-κP]-rhodium(I)

Michael Coetzee, Walter Purcell,* Hendrik G. Visser and Johan A. Venter

Department of Chemistry, University of the Free State, Bloemfontein 9300, South Africa
Correspondence e-mail: purcellw.sci@mail.uovs.ac.za

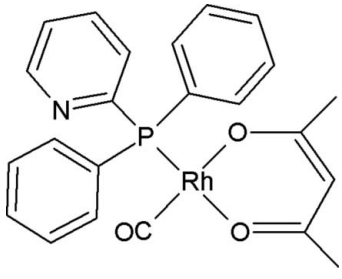
Received 13 November 2007; accepted 13 November 2007

Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.003 Å; R factor = 0.026; wR factor = 0.065; data-to-parameter ratio = 19.7.

The title complex, $[Rh(C_5H_7O_2)(C_{17}H_{14}NP)(CO)]$, adopts a square-planar geometry, with bite angles around the rhodium(I) centre varying between 85.28 (7) and 93.59 (8)°. The two phenyl rings and the one pyridine ring of the phosphine ligand are arranged in a face on/edge on orientation, with dihedral angles between the planes formed by these rings ranging between 66.55 (7) and 80.50 (7)°.

Related literature

For the synthesis of $[Rh(acac)(CO)_2]$, see: Leipoldt *et al.* (1978). For metal complexes of the ligand, see: Clarke *et al.* (2001); Cotton & Matusz (1988); Farr *et al.* (1983); Marsh (1997). For similar complexes, see: Brink *et al.* (2007*a*,*b*); Drommi *et al.* (1995); Wajda-Hermanowicz *et al.* (2006).



Experimental

Crystal data

$[Rh(C_5H_7O_2)(C_{17}H_{14}NP)(CO)]$
M_r = 493.29
Monoclinic, $P2_1/n$
a = 8.822 (5) Å
b = 17.782 (5) Å
c = 13.798 (5) Å
β = 103.027 (5)°
V = 2108.8 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.91 mm^{-1}
T = 150 K
0.19 × 0.18 × 0.06 mm

Data collection

Bruker Kappa APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.846, T_{max} = 0.947
24055 measured reflections
5248 independent reflections
4522 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.065
S = 1.05
5248 reflections
266 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

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).

The authors thank the Research Fund of the University of the Free State.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2373).

References

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.
Brandenburg, K. & Putz, H. (2005). *DIAMOND*. Release 3.0c. Crystal Impact GbR, Bonn, Germany.
Brink, A., Roodt, A. & Visser, H. G. (2007*a*). *Acta Cryst.* E**63**, m48–m50.
Brink, A., Roodt, A. & Visser, H. G. (2007*b*). *Acta Cryst.* E**63**, m2831–m2832.
Bruker (2004). *SAINT-Plus* (Version 7.12) and *SADABS* (Version 2004/1). Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2005). *APEX2*. Version 1.0-27. Bruker AXS Inc., Madison, Wisconsin, USA.
Clarke, M. L., Slawin, A. M. Z., Wheatley, M. V. & Woollins, J. D. (2001). *J. Chem. Soc. Dalton Trans.* pp. 3421–3423.
Cotton, F. A. & Matusz, M. (1988). *Inorg. Chim. Acta*, **143**, 45–46.
Drommi, D., Arena, C. G., Nicolo, F., Bruno, G. & Faraone, F. (1995). *J. Organomet. Chem.* **485**, 115–118.
Farr, J. P., Olmstead, M. M., Wood, F. E. & Balch, A. L. (1983). *J. Am. Chem. Soc.* **105**, 792–798.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Leipoldt J. G., Basson S. S., Bok L. D. C. and **&** Gerber T. I. A. (1978). *Inorg. Chim. Acta*, **26**, L35–L37.
Marsh, R. E. (1997). *Acta Cryst.* B**53**, 317–322.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Wajda-Hermanowicz, K., Ciunik, Z. & Kochel, A. (2006). *Inorg. Chem.* **45**, 3369–3372.

supplementary materials

Acta Cryst. (2007). E**63**, m3165 [**doi:10.1107/S1600536807058886**]

(Acetylacetonato-κ^2O,O')carbonyl[2-(diphenylphosphino)pyridine-κP]rhodium(I)

M. Coetzee, W. Purcell, H. G. Visser and J. A. Venter

Comment

The title compound (I) contains a rhodium(I) centre which is surrounded by two oxygen atoms of the acac ligand, a carbon atom of the carbonyl ligand and a phosphorous atom of the P(Pyr)$(Ph)_2$ ligand. The Rh1–C6 bonding distance is 1.810 (2) Å while the Rh1–O1 and Rh1–O2 distances are 2.0700 (15) and 2.0343 (15) Å respectively. The Rh1–P1 bonding distance is 2.2302 (8) Å.

The bite angles around the rhodium(I) centre vary between 85.28 (7) and 93.59 (8)°. All the bonding distances and angles fall in the expected range for similar complexes (Wajda-Hermanowicz *et al.* 2006, Drommi *et al.* 1995, Brink *et al.* 2007*a*, Brink *et al.* 2007*b*).

The P(Pyr)$(Ph)_2$ ligand is behaving as a monodentate ligand in (I), although several cases have been reported where it behaves as a bridging (Farr *et al.* 1983; Cotton & Matusz, 1988; Marsh, 1997) or a bidentate ligand (Clarke *et al.* 2001).

Experimental

[Rh(acac)$(CO)_2$] (Leipoldt *et al.*,1978) (0.0109 g, 0.042 mmol) was dissolved in methanol (5 ml). 2-(Diphenylphosphino)pyridine (0.0111 g, 0.042 mmol) was added to this solution. The solution was stirred until a yellow precipitate formed. The product, (I), was dissolved in 5 ml diethyl ether and filtered. Yellow crystals were obtained after 24 h in 70% yield. ^{31}P NMR ($CDCl_3$, p.p.m..): [δ, ^{1}J(Rh–P) = 177.09 Hz] IR ν(Rh—CO) 1963 cm^{-1}.

Refinement

The methyl, and aromatic H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with [C—H = 0.95–1.00 Å, and U_{iso}(H) = 1.5U_{eq}(C) and 1.2U_{eq}(C)]. The methyl H atoms were refined as a rigid rotor.

Figures

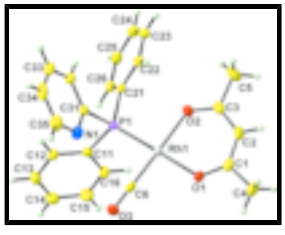


Fig. 1. View of (I) (50% probability displacement ellipsoids)

(Acetylacetonato-κ^2*O*,*O*')carbonyl[2- (diphenylphosphino)pyridine-κ*P*]rhodium(I)

Crystal data

$[Rh(C_5H_7O_2)(C_{17}H_{14}NP)(CO)]$	F_{000} = 1000
M_r = 493.29	D_x = 1.554 Mg m^{-3}
Monoclinic, *P*2(1)/*n*	Mo *K*α radiation λ = 0.71069 Å
Hall symbol: -P 2yn	Cell parameters from 8227 reflections
a = 8.822 (5) Å	θ = 2.3–28.2º
b = 17.782 (5) Å	μ = 0.91 mm^{-1}
c = 13.798 (5) Å	*T* = 150 K
β = 103.027 (5)º	Cuboid, yellow
V = 2108.8 (15) Å^3	0.19 × 0.18 × 0.06 mm
Z = 4	

Data collection

Bruker X8 APEXII 4K Kappa CCD diffractometer	R_{int} = 0.034
ω and φ scans	θ_{max} = 28.3º
Absorption correction: multi-scan (SADABS; Bruker, 2004)	θ_{min} = 2.5º
T_{min} = 0.846, T_{max} = 0.947	*h* = −11→11
24055 measured reflections	*k* = −23→23
5248 independent reflections	*l* = −18→18
4522 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0253P)^2 + 1.8318P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.026	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.065	$\Delta\rho_{max}$ = 0.46 e Å^{-3}
S = 1.05	$\Delta\rho_{min}$ = −0.39 e Å^{-3}
5248 reflections	Extinction correction: none
266 parameters	

Special details

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Rh1	0.413043 (18)	0.130551 (9)	0.345080 (11)	0.01678 (5)
P2	0.45125 (6)	0.07735 (3)	0.20616 (4)	0.01570 (10)
O3	0.5393 (2)	0.26775 (9)	0.27114 (12)	0.0325 (4)
O1	0.38355 (18)	0.18126 (8)	0.47462 (10)	0.0244 (3)
O2	0.32107 (17)	0.03483 (8)	0.38876 (10)	0.0234 (3)
C6	0.4920 (2)	0.21458 (12)	0.30102 (15)	0.0227 (4)
C26	0.2370 (2)	−0.00915 (12)	0.06995 (14)	0.0207 (4)
H26	0.2259	0.0327	0.0285	0.025*
N1	0.7625 (2)	0.09914 (12)	0.25525 (15)	0.0325 (4)
C16	0.2750 (2)	0.18390 (12)	0.08777 (15)	0.0233 (4)
H16	0.207	0.1765	0.1295	0.028*
C11	0.4094 (2)	0.14104 (11)	0.10007 (14)	0.0184 (4)
C21	0.3381 (2)	−0.00670 (11)	0.16307 (14)	0.0177 (4)
C25	0.1530 (3)	−0.07393 (13)	0.03899 (16)	0.0257 (5)
H25	0.0856	−0.0754	−0.0234	0.031*
C4	0.3242 (3)	0.20150 (15)	0.63005 (17)	0.0366 (6)
H4A	0.2795	0.2492	0.6065	0.055*
H4B	0.4284	0.2089	0.6681	0.055*
H4C	0.2628	0.1785	0.6712	0.055*
C13	0.4763 (3)	0.20638 (14)	−0.03672 (16)	0.0307 (5)
H13	0.5441	0.2141	−0.0785	0.037*
C23	0.2679 (3)	−0.13384 (12)	0.19316 (17)	0.0265 (5)
H23	0.2778	−0.1758	0.2344	0.032*
C32	0.6925 (2)	−0.02201 (12)	0.18125 (15)	0.0237 (4)
H32	0.6153	−0.0558	0.1518	0.028*
C12	0.5094 (3)	0.15155 (12)	0.03682 (16)	0.0238 (4)
H12	0.5982	0.122	0.0435	0.029*
C33	0.8482 (3)	−0.04076 (14)	0.19355 (17)	0.0318 (5)
H33	0.8767	−0.0875	0.1732	0.038*
C15	0.2423 (3)	0.23717 (14)	0.01422 (17)	0.0313 (5)
H15	0.1515	0.2654	0.0059	0.038*
C22	0.3524 (2)	−0.06947 (12)	0.22521 (16)	0.0223 (4)
H22	0.4187	−0.068	0.288	0.027*
C31	0.6531 (2)	0.04760 (11)	0.21339 (13)	0.0177 (4)
C34	0.9600 (3)	0.01062 (16)	0.23621 (17)	0.0351 (6)
H34	1.0651	−0.001	0.2455	0.042*
C35	0.9140 (3)	0.07956 (16)	0.26490 (18)	0.0363 (6)
H35	0.9904	0.1144	0.2922	0.044*
C14	0.3433 (3)	0.24935 (14)	−0.04791 (17)	0.0318 (5)
H14	0.3216	0.2863	−0.0968	0.038*
C5	0.2152 (3)	−0.05386 (14)	0.48055 (18)	0.0378 (6)
H5A	0.1784	−0.0764	0.4163	0.057*
H5C	0.1316	−0.0515	0.5146	0.057*
H5B	0.2984	−0.0836	0.5186	0.057*
C3	0.2732 (2)	0.02422 (13)	0.46823 (15)	0.0246 (4)

C24	0.1681 (3)	−0.13612 (13)	0.09948 (17)	0.0279 (5)
H24	0.1119	−0.1796	0.0779	0.034*
C1	0.3270 (3)	0.15107 (13)	0.54241 (15)	0.0250 (5)
C2	0.2724 (3)	0.07777 (14)	0.54181 (16)	0.0292 (5)
H2	0.2315	0.0631	0.5954	0.035*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Rh1	0.01685 (8)	0.01675 (8)	0.01773 (8)	−0.00021 (6)	0.00598 (6)	−0.00119 (6)
P2	0.0140 (2)	0.0158 (2)	0.0174 (2)	0.00126 (19)	0.00381 (18)	−0.00040 (18)
O3	0.0433 (10)	0.0230 (8)	0.0357 (9)	−0.0072 (7)	0.0184 (8)	−0.0026 (7)
O1	0.0303 (8)	0.0234 (8)	0.0216 (7)	0.0005 (6)	0.0107 (6)	−0.0030 (6)
O2	0.0252 (8)	0.0230 (8)	0.0225 (7)	−0.0038 (6)	0.0065 (6)	0.0010 (6)
C6	0.0253 (11)	0.0223 (11)	0.0224 (10)	−0.0011 (9)	0.0092 (8)	−0.0074 (8)
C26	0.0201 (10)	0.0229 (10)	0.0201 (9)	0.0001 (8)	0.0067 (8)	−0.0011 (8)
N1	0.0227 (10)	0.0404 (12)	0.0334 (10)	−0.0032 (9)	0.0045 (8)	−0.0052 (9)
C16	0.0193 (10)	0.0274 (11)	0.0235 (10)	0.0035 (9)	0.0055 (8)	0.0036 (8)
C11	0.0194 (10)	0.0172 (10)	0.0174 (9)	0.0007 (8)	0.0019 (7)	−0.0006 (7)
C21	0.0143 (9)	0.0184 (10)	0.0216 (9)	0.0005 (7)	0.0067 (7)	−0.0034 (7)
C25	0.0222 (11)	0.0316 (12)	0.0235 (10)	−0.0044 (9)	0.0053 (8)	−0.0068 (9)
C4	0.0488 (15)	0.0415 (15)	0.0224 (11)	0.0116 (12)	0.0141 (11)	−0.0012 (10)
C13	0.0347 (13)	0.0338 (13)	0.0264 (11)	0.0016 (10)	0.0126 (10)	0.0060 (9)
C23	0.0272 (11)	0.0194 (10)	0.0346 (11)	−0.0021 (9)	0.0107 (9)	0.0014 (9)
C32	0.0224 (10)	0.0237 (11)	0.0268 (10)	0.0034 (9)	0.0091 (8)	0.0019 (8)
C12	0.0236 (11)	0.0234 (11)	0.0253 (10)	0.0041 (8)	0.0075 (9)	0.0010 (8)
C33	0.0311 (12)	0.0352 (13)	0.0329 (12)	0.0154 (10)	0.0154 (10)	0.0079 (10)
C15	0.0263 (12)	0.0311 (13)	0.0346 (12)	0.0090 (10)	0.0030 (10)	0.0048 (10)
C22	0.0193 (10)	0.0225 (11)	0.0255 (10)	0.0013 (8)	0.0055 (8)	0.0000 (8)
C31	0.0153 (9)	0.0229 (10)	0.0153 (8)	0.0030 (8)	0.0041 (7)	0.0021 (7)
C34	0.0176 (11)	0.0613 (17)	0.0276 (11)	0.0098 (11)	0.0072 (9)	0.0128 (11)
C35	0.0215 (12)	0.0533 (16)	0.0319 (12)	−0.0077 (11)	0.0014 (10)	−0.0033 (11)
C14	0.0353 (13)	0.0288 (12)	0.0298 (11)	0.0039 (10)	0.0041 (10)	0.0094 (9)
C5	0.0445 (15)	0.0348 (14)	0.0333 (12)	−0.0097 (12)	0.0075 (11)	0.0100 (10)
C3	0.0193 (10)	0.0294 (12)	0.0237 (10)	−0.0011 (9)	0.0021 (8)	0.0077 (9)
C24	0.0254 (11)	0.0250 (11)	0.0356 (12)	−0.0068 (9)	0.0113 (9)	−0.0094 (9)
C1	0.0234 (11)	0.0316 (12)	0.0205 (10)	0.0082 (9)	0.0059 (8)	0.0017 (8)
C2	0.0332 (12)	0.0358 (13)	0.0211 (10)	0.0019 (10)	0.0112 (9)	0.0068 (9)

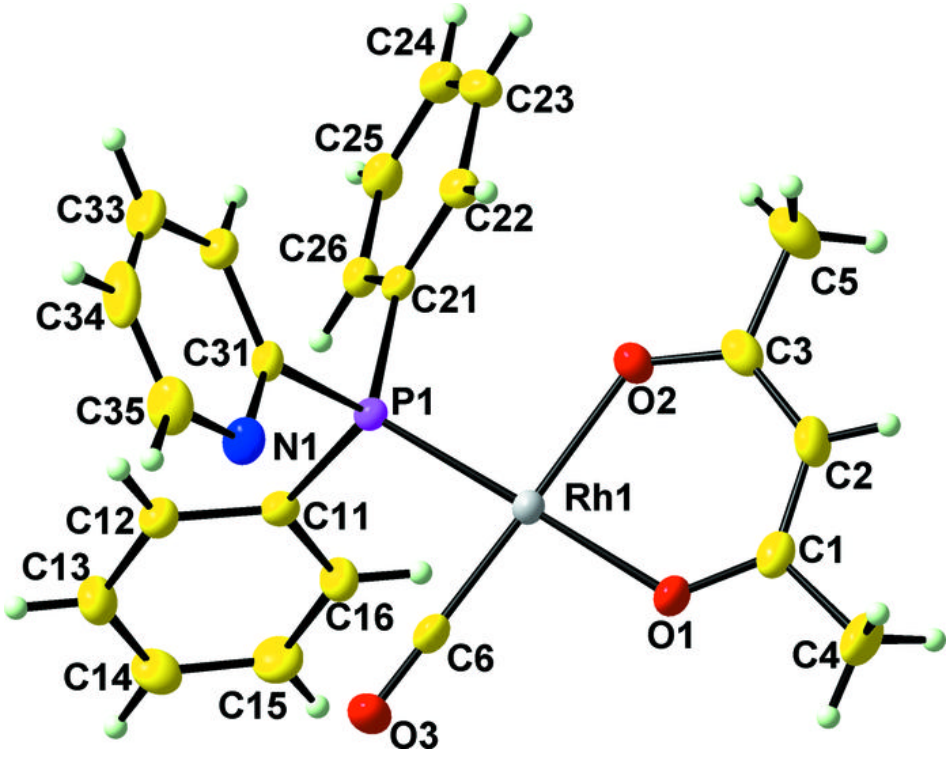
Geometric parameters (Å, °)

Rh1—C6	1.810 (2)	C13—C12	1.390 (3)
Rh1—O2	2.0345 (15)	C13—H13	0.93
Rh1—O1	2.0701 (15)	C23—C22	1.383 (3)
Rh1—P2	2.2303 (8)	C23—C24	1.391 (3)
P2—C21	1.821 (2)	C23—H23	0.93
P2—C11	1.821 (2)	C32—C31	1.385 (3)
P2—C31	1.839 (2)	C32—C33	1.386 (3)
O3—C6	1.147 (3)	C32—H32	0.93

O1—C1	1.274 (3)	C12—H12	0.93
O2—C3	1.275 (2)	C33—C34	1.376 (4)
C26—C25	1.384 (3)	C33—H33	0.93
C26—C21	1.390 (3)	C15—C14	1.386 (3)
C26—H26	0.93	C15—H15	0.93
N1—C35	1.359 (3)	C22—H22	0.93
N1—C31	1.361 (3)	C34—C35	1.377 (4)
C16—C15	1.371 (3)	C34—H34	0.93
C16—C11	1.387 (3)	C35—H35	0.93
C16—H16	0.93	C14—H14	0.93
C11—C12	1.386 (3)	C5—C3	1.502 (3)
C21—C22	1.396 (3)	C5—H5A	0.96
C25—C24	1.374 (3)	C5—H5C	0.96
C25—H25	0.93	C5—H5B	0.96
C4—C1	1.510 (3)	C3—C2	1.393 (3)
C4—H4A	0.96	C24—H24	0.93
C4—H4B	0.96	C1—C2	1.389 (3)
C4—H4C	0.96	C2—H2	0.93
C13—C14	1.379 (3)		
C6—Rh1—O2	177.68 (7)	C31—C32—H32	120.4
C6—Rh1—O1	93.62 (8)	C33—C32—H32	120.4
O2—Rh1—O1	88.42 (6)	C11—C12—C13	119.7 (2)
C6—Rh1—P2	85.26 (6)	C11—C12—H12	120.1
O2—Rh1—P2	92.71 (5)	C13—C12—H12	120.1
O1—Rh1—P2	178.43 (4)	C34—C33—C32	119.2 (2)
C21—P2—C11	104.54 (9)	C34—C33—H33	120.4
C21—P2—C31	103.56 (9)	C32—C33—H33	120.4
C11—P2—C31	103.93 (9)	C16—C15—C14	120.5 (2)
C21—P2—Rh1	116.79 (6)	C16—C15—H15	119.7
C11—P2—Rh1	112.61 (7)	C14—C15—H15	119.7
C31—P2—Rh1	114.05 (6)	C23—C22—C21	119.8 (2)
C1—O1—Rh1	126.64 (14)	C23—C22—H22	120.1
C3—O2—Rh1	127.61 (14)	C21—C22—H22	120.1
O3—C6—Rh1	178.4 (2)	N1—C31—C32	122.19 (19)
C25—C26—C21	119.9 (2)	N1—C31—P2	114.48 (15)
C25—C26—H26	120	C32—C31—P2	123.32 (16)
C21—C26—H26	120	C33—C34—C35	119.0 (2)
C35—N1—C31	117.2 (2)	C33—C34—H34	120.5
C15—C16—C11	120.1 (2)	C35—C34—H34	120.5
C15—C16—H16	119.9	N1—C35—C34	123.2 (2)
C11—C16—H16	119.9	N1—C35—H35	118.4
C12—C11—C16	119.75 (19)	C34—C35—H35	118.4
C12—C11—P2	123.20 (16)	C13—C14—C15	119.6 (2)
C16—C11—P2	116.91 (15)	C13—C14—H14	120.2
C26—C21—C22	119.56 (19)	C15—C14—H14	120.2
C26—C21—P2	121.18 (15)	C3—C5—H5A	109.5
C22—C21—P2	119.26 (15)	C3—C5—H5C	109.5
C24—C25—C26	120.7 (2)	H5A—C5—H5C	109.5
C24—C25—H25	119.7	C3—C5—H5B	109.5

C26—C25—H25	119.7	H5A—C5—H5B	109.5
C1—C4—H4A	109.5	H5C—C5—H5B	109.5
C1—C4—H4B	109.5	O2—C3—C2	125.6 (2)
H4A—C4—H4B	109.5	O2—C3—C5	114.9 (2)
C1—C4—H4C	109.5	C2—C3—C5	119.4 (2)
H4A—C4—H4C	109.5	C25—C24—C23	119.7 (2)
H4B—C4—H4C	109.5	C25—C24—H24	120.1
C14—C13—C12	120.2 (2)	C23—C24—H24	120.1
C14—C13—H13	119.9	O1—C1—C2	125.7 (2)
C12—C13—H13	119.9	O1—C1—C4	114.7 (2)
C22—C23—C24	120.3 (2)	C2—C1—C4	119.6 (2)
C22—C23—H23	119.9	C1—C2—C3	125.94 (19)
C24—C23—H23	119.9	C1—C2—H2	117
C31—C32—C33	119.3 (2)	C3—C2—H2	117

Fig. 1


C24
C23
C25
C22
C33
C26
C21
C5
C34
C31
C3
C35
P1
O2
N1
C2
Rh1
C12
C11
C1
C13
C16
O1
C6
C4
C14
C15
O3