

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

trans-Carbonylchloridobis[tris(4-chlorophenyl)phosphane]rhodium(I) acetone monosolvate

Andrew R. Burgoyne, Reinout Meijboom,* Alfred Muller and Bernard O. Omondi

Research Centre for Synthesis and Catalysis, Department of Chemistry, University of Johannesburg, PO Box 524 Auckland Park, Johannesburg, 2006, South Africa

Correspondence e-mail: rmeijboom@uj.ac.za

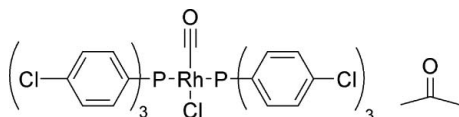
Received 22 September 2010; accepted 5 October 2010

Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.026; wR factor = 0.069; data-to-parameter ratio = 21.3.

The title compound, *trans*-[RhCl(C₁₈H₁₂Cl₃P)₂(CO)]·C₃H₆O, contains an Rh(I) atom in a distorted square-planar coordination with a P—Rh—P angle of 175.27 (2)° and Rh—P bond lengths of 2.3127 (4) and 2.3219 (4) Å. The rhodium complexes link each other through weak intermolecular contacts between the acetone methyl groups and the carbonyl O atom. Interactions between the acetone solvent molecule and the Cl—Rh unit results in a reduced P—Rh—Cl angle of 86.675 (15)°.

Related literature

For a review of rhodium Vaska {*trans*-[RhCl(CO)(PR₃)₂]} compounds, see: Roodt *et al.* (2003). For related compounds, see: Angoletta (1959); Vaska & Di Luzio (1961); Chen *et al.* (1991); Kuwabara & Bau (1994); Otto *et al.* (2000); Otto (2001); Meijboom *et al.* (2005).



Experimental

Crystal data

[RhCl(C₁₈H₁₂Cl₃P)₂(CO)]·C₃H₆O

$M_r = 955.64$

Triclinic, $P\bar{1}$

$a = 10.6130$ (7) Å

$b = 12.7970$ (8) Å

$c = 16.7470$ (11) Å

$\alpha = 71.631$ (1)°

$\beta = 81.742$ (1)°

$\gamma = 68.537$ (1)°

$V = 2007.9$ (2) Å³

$Z = 2$

Mo $K\alpha$ radiation

$\mu = 1.01$ mm⁻¹

$T = 100$ K

$0.37 \times 0.17 \times 0.07$ mm

Data collection

Bruker X8 APEXII 4K KappaCCD diffractometer

Absorption correction: multi-scan (SADABS; Bruker, 2007)

$T_{\min} = 0.707$, $T_{\max} = 0.933$

25954 measured reflections

10031 independent reflections

9160 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.023$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.026$

$wR(F^2) = 0.069$

$S = 1.04$

10031 reflections

471 parameters

4 restraints

H-atom parameters constrained

$\Delta\rho_{\text{max}} = 1.17$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.69$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

C37—O1	1.1420 (18)	P2—Rh1	2.3219 (4)
C37—Rh1	1.8177 (13)	Cl1—Rh1	2.3611 (4)
P1—Rh1	2.3127 (4)		
O1—C37—Rh1	179.52 (16)	P1—Rh1—Cl1	86.675 (15)
P1—Rh1—P2	175.268 (15)	P2—Rh1—Cl1	90.348 (15)
C37—Rh1—Cl1	178.21 (5)		

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C15—H15 ⁱ ···O2 ⁱ	0.95	2.55	3.492 (3)	170
C21—H21 ⁱ ···O2 ⁱⁱ	0.95	2.34	3.267 (3)	165
C24—H24 ⁱ ···Cl5 ⁱⁱⁱ	0.95	2.80	3.4734 (19)	129
C30—H30 ⁱ ···Cl4 ^{iv}	0.95	2.76	3.575 (2)	145
C35—H35 ⁱ ···O1 ^v	0.95	2.47	3.384 (2)	161
C40—H40B ⁱ ···Cl1	0.98	2.79	3.738 (3)	162

Symmetry codes: (i) $x, y + 1, z$; (ii) $x + 1, y, z$; (iii) $-x + 1, -y, -z + 1$; (iv) $-x + 2, -y, -z + 1$; (v) $-x + 2, -y + 1, -z + 1$.

Data collection: APEX2 (Bruker, 2007); cell refinement: SAINT-Plus (Bruker, 2007); data reduction: SAINT-Plus and XPREP (Bruker, 2007); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: DIAMOND (Brandenburg & Putz, 2005) and ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999).

ARB thanks the Research Academy for Undergraduates, University of Johannesburg, for financial support. Financial assistance from the South African National Research Foundation and the University of Johannesburg is also gratefully acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2294).

References

- Angoletta, M. (1959). *Gazz. Chim. Ital.* **89**, 2359–2361.
- Brandenburg, K. & Putz, H. (2005). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
- Bruker (2007). APEX2, SAINT-Plus and SADABS. BrukerAXS Inc, Madison, Wisconsin, USA.
- Chen, Y.-J., Wang, J.-C. & Wang, Y. (1991). *Acta Cryst.* **C47**, 2441–2442.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.

- Kuwabara, E. & Bau, R. (1994). *Acta Cryst.* **C50**, 1409–1411.
Meijboom, R., Muller, A. & Roodt, A. (2005). *Acta Cryst.* **E61**, m1283–m1285.
Otto, S. (2001). *Acta Cryst.* **C57**, 793–795.
Otto, S., Roodt, A. & Smith, J. (2000). *Inorg. Chim. Acta*, **303**, 295–299.
Roodt, A., Otto, S. & Steyl, G. (2003). *Coord. Chem. Rev.* **245**, 121–137.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
Vaska, L. & Di Luzio, J. W. (1961). *J. Am. Chem. Soc.* **83**, 2784–2785.

supplementary materials

Acta Cryst. (2010). E66, m1380-m1381 [doi:10.1107/S1600536810039814]

***trans*-Carbonylchloridobis[tris(4-chlorophenyl)phosphane]rhodium(I) acetone monosolvate**

A. R. Burgoyne, R. Meijboom, A. Muller and B. O. Omondi

Comment

The original Vaska complex, *trans*-[IrCl(CO)(PPh₃)₂], was first reported in 1959 (Angoletta, 1959), but later correctly formulated by Vaska in 1961 (Vaska & Di Luzio, 1961). This class of symmetrical square-planar complexes often crystallizes with the metal atom on a crystallographic centre of symmetry, thus imposing a disordered packing arrangement (Otto, 2001; Otto *et al.*, 2000; Chen *et al.*, 1991; Kuwabara & Bau, 1994). We report the title compound as one of the few crystallographic examples of this group which does not show disorder along the carbonyl/chloro axis.

Compound (I) crystallizes in a triclinic space group. The crystal structure of *trans*-[RhCl(CO){P(4-ClC₆H₄)₃}₂] (Fig. 1) shows the expected square planar geometry with the phosphine ligands *trans* to each other.

As seen in Fig. 1 and Table 1, the complex shows a non-equally spaced square planar coordination centre with the P—Rh—P angle being 175.27 (2) °. The ¹J(Rh—P) coupling of 128 Hz is in agreement with the coupling constants for other rhodium Vaska type complexes of this nature (Meijboom *et al.*, 2005).

Table 1 shows the angles C37—Rh—P1 and C37—Rh—P2 are nearly equal, while P2—Rh—Cl is 90.348 (15) ° and P1—Rh—Cl is only 86.675 (15) °. This inequality around the chlorine's displacement is interesting as the most linear hydrogen bond involving Cl1 is between C40—H40···Cl1 (162 °, Table 2) pulling it towards C40—H40 and thus decreasing the angle of P1—Rh—Cl. Additionally, the rhodium complexes link each other *via* weak intermolecular C—H···O and C—H···Cl hydrogen bonds (Table 2). The weak interactions between the acetone solvent molecule and the Rh—Cl unit resulted in a reduced P—Rh—Cl angle of 86.675 (15)°.

The ν(CO) stretching frequency of compound (1) is 1985 cm⁻¹ which is higher than that of [Rh(CO)Cl{P(C₆H₅)₃}₂], the unchlorinated version, which was reported by Roodt (Roodt *et al.*, 2003) as 1979 cm⁻¹. The increase is due to the moderate electron withdrawing capabilities of the *para*-substituted chlorine on the phenyl rings.

Experimental

To a solution of [Rh₂(CO)₄Cl₂] (19.7 mg, 0.051 mmol) in acetone (3 cm³) was added 4 equivalents of a solution of P(4-ClC₆H₄)₃ ligand (73.7 mg, 0.203 mmol) in acetone (2 cm³) while stirring. Immediately the effervescence of a gas, presumably CO, was observed. The solvent was evaporated in a round bottomed flask overnight yielding 62% of the title compound (28.2 mg, 0.0314 mmol). IR (cm⁻¹): 629, 705, 746, 815, 1012, 1082, 1120, 1183, 1226, 1363, 1387, 1479, 1560, 1577, 1638, 1706, 1985 (CO). ¹³C NMR (δ /p.p.m., CDCl₃): 139 (1 C, Ph), 137 (1 C, Ph), 135 (1 C, Ph), 133 (1 C, Ph), 130 (4 C, Ph), 129 (1 C, CO), 129 (1 C, Ph), 128 (1 C, Ph). ³¹P NMR (δ /p.p.m., CDCl₃): 27.56 [¹J(Rh—P) = 128 Hz].

Refinement

The methyl and aromatic H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.95–0.98 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ and $1.2U_{\text{eq}}(\text{C})$, respectively. The methyl groups were generated to fit the difference electron density and the groups were then refined as rigid rotors.

Figures

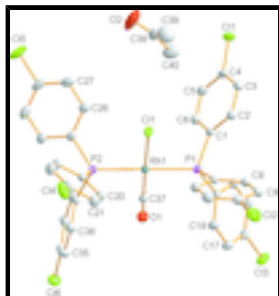


Fig. 1. View of (I) (50% probability displacement ellipsoids) with H atoms omitted for clarity. The carbon atoms have been numbered sequentially and some labels have been omitted for clarity.

trans-Carbonylchloridobis[tris(4-chlorophenyl)phosphane]rhodium(I) acetone monosolvate

Crystal data

$[\text{RhCl}(\text{C}_{18}\text{H}_{12}\text{Cl}_3\text{P})_2(\text{CO})] \cdot \text{C}_3\text{H}_6\text{O}$

$M_r = 955.64$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 10.6130$ (7) Å

$b = 12.7970$ (8) Å

$c = 16.7470$ (11) Å

$\alpha = 71.631$ (1)°

$\beta = 81.742$ (1)°

$\gamma = 68.537$ (1)°

$V = 2007.9$ (2) Å³

$Z = 2$

$F(000) = 960$

$D_x = 1.581$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 25954 reflections

$\theta = 1.8$ – 28.4 °

$\mu = 1.01$ mm⁻¹

$T = 100$ K

Block, yellow

$0.37 \times 0.17 \times 0.07$ mm

Data collection

Bruker X8 APEXII 4K KappaCCD diffractometer

graphite

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2007)

$T_{\text{min}} = 0.707$, $T_{\text{max}} = 0.933$

25954 measured reflections

10031 independent reflections

9160 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.023$

$\theta_{\text{max}} = 28.4$ °, $\theta_{\text{min}} = 1.8$ °

$h = -14 \rightarrow 12$

$k = -16 \rightarrow 17$

$l = -21 \rightarrow 22$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.026$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.069$	H-atom parameters constrained
$S = 1.04$	$w = 1/[\sigma^2(F_o^2) + (0.0302P)^2 + 1.7506P]$
10031 reflections	where $P = (F_o^2 + 2F_c^2)/3$
471 parameters	$(\Delta/\sigma)_{\max} = 0.009$
4 restraints	$\Delta\rho_{\max} = 1.17 \text{ e } \text{\AA}^{-3}$
	$\Delta\rho_{\min} = -0.69 \text{ e } \text{\AA}^{-3}$

Special details

Experimental. The intensity data was collected on a Bruker X8 Apex II CCD diffractometer using an exposure time of 10 s/frame. A total of 1895 frames were collected with a frame width of 0.5° covering up to $\theta = 28.38^\circ$ with 99.9% completeness accomplished.

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.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.45279 (16)	0.41780 (13)	0.86206 (10)	0.0164 (3)
C2	0.41530 (18)	0.37176 (15)	0.94513 (11)	0.0210 (3)
H2	0.4811	0.3368	0.9874	0.025*
C3	0.28161 (19)	0.37668 (16)	0.96678 (12)	0.0245 (4)
H3	0.2559	0.3451	1.0234	0.029*
C4	0.18729 (17)	0.42829 (16)	0.90434 (12)	0.0232 (3)
C5	0.22084 (18)	0.47737 (16)	0.82210 (12)	0.0245 (4)
H5	0.1539	0.5149	0.7805	0.029*
C6	0.35413 (18)	0.47109 (15)	0.80104 (11)	0.0214 (3)
H6	0.3787	0.5035	0.7443	0.026*
C7	0.74075 (16)	0.32026 (14)	0.90731 (10)	0.0171 (3)
C8	0.73223 (17)	0.35235 (15)	0.98114 (11)	0.0198 (3)
H8	0.6603	0.4195	0.9892	0.024*
C9	0.82786 (18)	0.28702 (17)	1.04254 (11)	0.0245 (4)
H9	0.8209	0.3078	1.0931	0.029*
C10	0.93422 (19)	0.19046 (17)	1.02877 (12)	0.0261 (4)

supplementary materials

C11	0.94494 (18)	0.15729 (15)	0.95637 (12)	0.0249 (4)
H11	1.0181	0.091	0.9481	0.03*
C12	0.84757 (17)	0.22195 (15)	0.89602 (11)	0.0212 (3)
H12	0.8536	0.1991	0.8464	0.025*
C13	0.62608 (16)	0.55598 (13)	0.81044 (10)	0.0161 (3)
C14	0.51081 (17)	0.64893 (15)	0.82071 (11)	0.0205 (3)
H14	0.4265	0.6367	0.8359	0.025*
C15	0.51770 (19)	0.75957 (15)	0.80891 (12)	0.0244 (4)
H15	0.4384	0.823	0.8147	0.029*
C16	0.6411 (2)	0.77582 (15)	0.78876 (11)	0.0232 (3)
C17	0.7573 (2)	0.68537 (16)	0.77805 (13)	0.0263 (4)
H17	0.8416	0.698	0.7638	0.032*
C18	0.74917 (18)	0.57584 (15)	0.78836 (12)	0.0230 (3)
H18	0.8283	0.5137	0.7803	0.028*
C19	0.86583 (16)	0.11855 (14)	0.62309 (10)	0.0173 (3)
C20	0.96226 (18)	0.10485 (16)	0.67746 (11)	0.0232 (3)
H20	0.9571	0.1691	0.6962	0.028*
C21	1.06604 (19)	-0.00186 (17)	0.70460 (12)	0.0276 (4)
H21	1.133	-0.0108	0.7406	0.033*
C22	1.06923 (19)	-0.09470 (16)	0.67782 (11)	0.0256 (4)
C23	0.9774 (2)	-0.08305 (16)	0.62225 (12)	0.0266 (4)
H23	0.9838	-0.1473	0.6031	0.032*
C24	0.87527 (19)	0.02477 (15)	0.59474 (12)	0.0236 (3)
H24	0.8115	0.0343	0.5563	0.028*
C25	0.59960 (17)	0.22421 (14)	0.55503 (11)	0.0196 (3)
C26	0.49901 (18)	0.19286 (16)	0.60992 (12)	0.0242 (4)
H26	0.4953	0.192	0.6671	0.029*
C27	0.4036 (2)	0.16278 (17)	0.58096 (14)	0.0302 (4)
H27	0.335	0.1412	0.6181	0.036*
C28	0.4105 (2)	0.16491 (16)	0.49772 (14)	0.0304 (4)
C29	0.5097 (2)	0.19354 (18)	0.44249 (13)	0.0312 (4)
H29	0.5133	0.1929	0.3856	0.037*
C30	0.60475 (19)	0.22352 (17)	0.47133 (12)	0.0259 (4)
H30	0.6738	0.2437	0.4338	0.031*
C31	0.79052 (17)	0.34837 (14)	0.50272 (10)	0.0183 (3)
C32	0.69971 (18)	0.44776 (16)	0.45032 (11)	0.0223 (3)
H32	0.6058	0.4585	0.4557	0.027*
C33	0.74499 (19)	0.53101 (16)	0.39047 (11)	0.0248 (4)
H33	0.6828	0.5983	0.3551	0.03*
C34	0.8822 (2)	0.51408 (16)	0.38325 (11)	0.0246 (4)
C35	0.97511 (19)	0.41477 (17)	0.43215 (12)	0.0261 (4)
H35	1.0692	0.4032	0.425	0.031*
C36	0.92848 (18)	0.33212 (15)	0.49190 (11)	0.0220 (3)
H36	0.9915	0.2637	0.5258	0.026*
C37	0.69720 (17)	0.47391 (12)	0.63949 (10)	0.0180 (3)
P1	0.62373 (4)	0.40975 (3)	0.82264 (3)	0.01420 (8)
P2	0.72683 (4)	0.25790 (4)	0.59485 (3)	0.01550 (8)
Cl1	0.63216 (5)	0.17010 (4)	0.79542 (3)	0.02401 (9)
Cl2	1.06102 (5)	0.11330 (5)	1.10165 (3)	0.04090 (13)

Cl3	0.65127 (5)	0.91247 (4)	0.77695 (3)	0.03312 (11)
Cl4	1.18984 (6)	-0.23221 (5)	0.71789 (3)	0.03995 (13)
Cl5	0.28900 (6)	0.13135 (5)	0.46052 (4)	0.04566 (15)
Cl6	0.93897 (6)	0.62136 (4)	0.31201 (3)	0.03453 (11)
Cl7	0.02340 (5)	0.42625 (5)	0.92807 (4)	0.03486 (11)
Rh1	0.668804 (12)	0.340979 (10)	0.705687 (7)	0.01450 (4)
O1	0.71508 (14)	0.55764 (11)	0.59840 (8)	0.0272 (3)
C38	0.3033 (2)	0.03136 (18)	0.88771 (14)	0.0311 (4)
C39	0.3764 (3)	-0.0610 (2)	0.96104 (18)	0.0542 (7)
H39A	0.3645	-0.1349	0.9651	0.081*
H39B	0.34	-0.038	1.0126	0.081*
H39C	0.473	-0.071	0.9539	0.081*
C40	0.2919 (3)	0.1538 (2)	0.87945 (19)	0.0463 (6)
H40A	0.2352	0.2075	0.8318	0.07*
H40B	0.3823	0.1605	0.8699	0.07*
H40C	0.2506	0.174	0.9312	0.07*
O2	0.2549 (2)	0.0074 (2)	0.83829 (16)	0.0709 (7)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0168 (7)	0.0131 (7)	0.0212 (8)	-0.0044 (6)	-0.0005 (6)	-0.0083 (6)
C2	0.0219 (8)	0.0212 (8)	0.0215 (8)	-0.0077 (6)	-0.0018 (6)	-0.0073 (6)
C3	0.0255 (9)	0.0272 (9)	0.0248 (9)	-0.0129 (7)	0.0046 (7)	-0.0105 (7)
C4	0.0173 (8)	0.0227 (8)	0.0354 (10)	-0.0090 (6)	0.0045 (7)	-0.0159 (7)
C5	0.0202 (8)	0.0237 (8)	0.0309 (9)	-0.0053 (7)	-0.0050 (7)	-0.0104 (7)
C6	0.0213 (8)	0.0206 (8)	0.0222 (8)	-0.0066 (6)	-0.0021 (6)	-0.0058 (6)
C7	0.0165 (7)	0.0148 (7)	0.0194 (7)	-0.0044 (6)	-0.0028 (6)	-0.0044 (6)
C8	0.0182 (8)	0.0202 (8)	0.0219 (8)	-0.0061 (6)	-0.0002 (6)	-0.0079 (6)
C9	0.0226 (8)	0.0317 (9)	0.0203 (8)	-0.0103 (7)	-0.0015 (7)	-0.0070 (7)
C10	0.0211 (8)	0.0276 (9)	0.0237 (9)	-0.0070 (7)	-0.0065 (7)	0.0019 (7)
C11	0.0205 (8)	0.0184 (8)	0.0292 (9)	-0.0015 (6)	-0.0027 (7)	-0.0031 (7)
C12	0.0206 (8)	0.0173 (8)	0.0245 (8)	-0.0042 (6)	-0.0014 (6)	-0.0069 (6)
C13	0.0200 (8)	0.0129 (7)	0.0169 (7)	-0.0055 (6)	-0.0023 (6)	-0.0056 (6)
C14	0.0195 (8)	0.0169 (8)	0.0260 (8)	-0.0048 (6)	-0.0023 (6)	-0.0083 (6)
C15	0.0260 (9)	0.0147 (8)	0.0318 (9)	-0.0019 (6)	-0.0047 (7)	-0.0100 (7)
C16	0.0351 (10)	0.0133 (7)	0.0237 (8)	-0.0102 (7)	-0.0065 (7)	-0.0042 (6)
C17	0.0264 (9)	0.0222 (8)	0.0346 (10)	-0.0128 (7)	-0.0008 (7)	-0.0087 (7)
C18	0.0203 (8)	0.0176 (8)	0.0326 (9)	-0.0061 (6)	0.0000 (7)	-0.0099 (7)
C19	0.0166 (7)	0.0168 (7)	0.0177 (7)	-0.0042 (6)	-0.0005 (6)	-0.0058 (6)
C20	0.0241 (9)	0.0229 (8)	0.0233 (8)	-0.0051 (7)	-0.0047 (7)	-0.0093 (7)
C21	0.0220 (9)	0.0317 (10)	0.0247 (9)	-0.0018 (7)	-0.0065 (7)	-0.0083 (7)
C22	0.0230 (9)	0.0217 (8)	0.0202 (8)	0.0029 (7)	-0.0002 (7)	-0.0029 (7)
C23	0.0302 (10)	0.0183 (8)	0.0298 (9)	-0.0023 (7)	-0.0027 (7)	-0.0112 (7)
C24	0.0248 (9)	0.0198 (8)	0.0272 (9)	-0.0039 (7)	-0.0068 (7)	-0.0098 (7)
C25	0.0176 (8)	0.0165 (7)	0.0251 (8)	-0.0024 (6)	-0.0053 (6)	-0.0085 (6)
C26	0.0218 (8)	0.0206 (8)	0.0311 (9)	-0.0062 (7)	-0.0041 (7)	-0.0084 (7)
C27	0.0218 (9)	0.0230 (9)	0.0469 (12)	-0.0078 (7)	-0.0057 (8)	-0.0093 (8)

supplementary materials

C28	0.0251 (9)	0.0180 (8)	0.0511 (12)	-0.0031 (7)	-0.0179 (8)	-0.0118 (8)
C29	0.0332 (10)	0.0286 (10)	0.0345 (10)	-0.0045 (8)	-0.0150 (8)	-0.0138 (8)
C30	0.0242 (9)	0.0276 (9)	0.0281 (9)	-0.0059 (7)	-0.0055 (7)	-0.0125 (7)
C31	0.0203 (8)	0.0183 (7)	0.0163 (7)	-0.0049 (6)	0.0003 (6)	-0.0071 (6)
C32	0.0195 (8)	0.0233 (8)	0.0207 (8)	-0.0032 (6)	0.0001 (6)	-0.0069 (7)
C33	0.0284 (9)	0.0199 (8)	0.0202 (8)	-0.0021 (7)	-0.0017 (7)	-0.0049 (7)
C34	0.0319 (10)	0.0213 (8)	0.0213 (8)	-0.0120 (7)	0.0031 (7)	-0.0054 (7)
C35	0.0226 (9)	0.0268 (9)	0.0292 (9)	-0.0111 (7)	0.0015 (7)	-0.0061 (7)
C36	0.0209 (8)	0.0203 (8)	0.0234 (8)	-0.0061 (6)	-0.0017 (6)	-0.0050 (7)
C37	0.0184 (7)	0.0166 (7)	0.0203 (7)	-0.0053 (6)	-0.0007 (6)	-0.0075 (6)
P1	0.01491 (18)	0.01144 (17)	0.01693 (18)	-0.00364 (14)	-0.00065 (14)	-0.00590 (14)
P2	0.01585 (19)	0.01487 (18)	0.01667 (19)	-0.00427 (15)	-0.00100 (14)	-0.00663 (15)
Cl1	0.0373 (2)	0.01797 (18)	0.02145 (19)	-0.01497 (17)	0.00302 (16)	-0.00717 (15)
Cl2	0.0270 (2)	0.0523 (3)	0.0291 (2)	-0.0029 (2)	-0.01223 (19)	-0.0002 (2)
Cl3	0.0448 (3)	0.0168 (2)	0.0426 (3)	-0.01495 (19)	-0.0064 (2)	-0.00755 (18)
Cl4	0.0390 (3)	0.0304 (2)	0.0270 (2)	0.0134 (2)	-0.0045 (2)	-0.00510 (19)
Cl5	0.0371 (3)	0.0340 (3)	0.0742 (4)	-0.0102 (2)	-0.0292 (3)	-0.0170 (3)
Cl6	0.0432 (3)	0.0276 (2)	0.0313 (2)	-0.0178 (2)	0.0031 (2)	-0.00148 (19)
Cl7	0.0213 (2)	0.0458 (3)	0.0463 (3)	-0.0170 (2)	0.00739 (19)	-0.0223 (2)
Rh1	0.01681 (7)	0.01199 (6)	0.01609 (6)	-0.00515 (4)	0.00030 (4)	-0.00597 (4)
O1	0.0338 (7)	0.0195 (6)	0.0280 (7)	-0.0111 (5)	-0.0011 (5)	-0.0040 (5)
C38	0.0233 (9)	0.0279 (10)	0.0451 (12)	-0.0039 (7)	-0.0021 (8)	-0.0200 (9)
C39	0.0591 (17)	0.0391 (13)	0.0492 (15)	-0.0084 (12)	-0.0019 (12)	-0.0020 (11)
C40	0.0412 (13)	0.0286 (11)	0.0677 (17)	-0.0111 (10)	-0.0042 (12)	-0.0116 (11)
O2	0.0511 (12)	0.0709 (14)	0.1097 (18)	0.0001 (10)	-0.0314 (12)	-0.0635 (14)

Geometric parameters (Å, °)

C1—C2	1.392 (2)	C22—C23	1.380 (3)
C1—C6	1.397 (2)	C22—Cl4	1.7434 (18)
C1—P1	1.8165 (17)	C23—C24	1.393 (2)
C2—C3	1.396 (2)	C23—H23	0.95
C2—H2	0.95	C24—H24	0.95
C3—C4	1.383 (3)	C25—C26	1.395 (3)
C3—H3	0.95	C25—C30	1.398 (2)
C4—C5	1.377 (3)	C25—P2	1.8221 (17)
C4—Cl7	1.7356 (18)	C26—C27	1.397 (3)
C5—C6	1.387 (2)	C26—H26	0.95
C5—H5	0.95	C27—C28	1.378 (3)
C6—H6	0.95	C27—H27	0.95
C7—C12	1.396 (2)	C28—C29	1.373 (3)
C7—C8	1.402 (2)	C28—Cl5	1.7438 (19)
C7—P1	1.8183 (17)	C29—C30	1.392 (3)
C8—C9	1.387 (2)	C29—H29	0.95
C8—H8	0.95	C30—H30	0.95
C9—C10	1.392 (3)	C31—C36	1.395 (2)
C9—H9	0.95	C31—C32	1.400 (2)
C10—C11	1.383 (3)	C31—P2	1.8235 (17)
C10—Cl2	1.7366 (19)	C32—C33	1.391 (2)

C11—C12	1.386 (2)	C32—H32	0.95
C11—H11	0.95	C33—C34	1.385 (3)
C12—H12	0.95	C33—H33	0.95
C13—C14	1.394 (2)	C34—C35	1.384 (3)
C13—C18	1.397 (2)	C34—Cl6	1.7426 (18)
C13—P1	1.8265 (16)	C35—C36	1.392 (2)
C14—C15	1.394 (2)	C35—H35	0.95
C14—H14	0.95	C36—H36	0.95
C15—C16	1.378 (3)	C37—O1	1.1420 (18)
C15—H15	0.95	C37—Rh1	1.8177 (13)
C16—C17	1.384 (3)	P1—Rh1	2.3127 (4)
C16—Cl3	1.7387 (17)	P2—Rh1	2.3219 (4)
C17—C18	1.390 (2)	Cl1—Rh1	2.3611 (4)
C17—H17	0.95	C38—O2	1.200 (3)
C18—H18	0.95	C38—C39	1.481 (3)
C19—C24	1.391 (2)	C38—C40	1.490 (3)
C19—C20	1.394 (2)	C39—H39A	0.98
C19—P2	1.8231 (17)	C39—H39B	0.98
C20—C21	1.392 (3)	C39—H39C	0.98
C20—H20	0.95	C40—H40A	0.98
C21—C22	1.384 (3)	C40—H40B	0.98
C21—H21	0.95	C40—H40C	0.98
C2—C1—C6	119.00 (15)	C23—C24—H24	119.7
C2—C1—P1	125.78 (13)	C26—C25—C30	119.21 (17)
C6—C1—P1	115.16 (12)	C26—C25—P2	119.51 (14)
C1—C2—C3	120.45 (16)	C30—C25—P2	121.21 (14)
C1—C2—H2	119.8	C25—C26—C27	120.16 (18)
C3—C2—H2	119.8	C25—C26—H26	119.9
C4—C3—C2	118.86 (17)	C27—C26—H26	119.9
C4—C3—H3	120.6	C28—C27—C26	119.00 (19)
C2—C3—H3	120.6	C28—C27—H27	120.5
C5—C4—C3	121.87 (16)	C26—C27—H27	120.5
C5—C4—Cl7	118.27 (15)	C29—C28—C27	122.19 (18)
C3—C4—Cl7	119.80 (14)	C29—C28—Cl5	118.59 (17)
C4—C5—C6	118.82 (17)	C27—C28—Cl5	119.22 (17)
C4—C5—H5	120.6	C28—C29—C30	118.81 (19)
C6—C5—H5	120.6	C28—C29—H29	120.6
C5—C6—C1	120.96 (16)	C30—C29—H29	120.6
C5—C6—H6	119.5	C29—C30—C25	120.61 (19)
C1—C6—H6	119.5	C29—C30—H30	119.7
C12—C7—C8	119.10 (15)	C25—C30—H30	119.7
C12—C7—P1	119.17 (13)	C36—C31—C32	118.67 (16)
C8—C7—P1	121.57 (12)	C36—C31—P2	120.49 (13)
C9—C8—C7	120.63 (16)	C32—C31—P2	119.87 (13)
C9—C8—H8	119.7	C33—C32—C31	120.96 (17)
C7—C8—H8	119.7	C33—C32—H32	119.5
C8—C9—C10	118.82 (17)	C31—C32—H32	119.5
C8—C9—H9	120.6	C34—C33—C32	118.87 (17)
C10—C9—H9	120.6	C34—C33—H33	120.6

supplementary materials

C11—C10—C9	121.59 (17)	C32—C33—H33	120.6
C11—C10—Cl2	118.84 (15)	C35—C34—C33	121.55 (16)
C9—C10—Cl2	119.51 (15)	C35—C34—Cl6	119.26 (15)
C10—C11—Cl2	119.16 (17)	C33—C34—Cl6	119.19 (14)
C10—C11—H11	120.4	C34—C35—C36	119.00 (17)
C12—C11—H11	120.4	C34—C35—H35	120.5
C11—C12—C7	120.69 (17)	C36—C35—H35	120.5
C11—C12—H12	119.7	C35—C36—C31	120.89 (17)
C7—C12—H12	119.7	C35—C36—H36	119.6
C14—C13—C18	118.59 (15)	C31—C36—H36	119.6
C14—C13—P1	123.20 (13)	O1—C37—Rh1	179.52 (16)
C18—C13—P1	118.21 (12)	C1—P1—C7	108.94 (8)
C13—C14—C15	120.87 (16)	C1—P1—C13	103.78 (7)
C13—C14—H14	119.6	C7—P1—C13	101.87 (7)
C15—C14—H14	119.6	C1—P1—Rh1	108.60 (5)
C16—C15—C14	119.16 (16)	C7—P1—Rh1	114.06 (5)
C16—C15—H15	120.4	C13—P1—Rh1	118.80 (5)
C14—C15—H15	120.4	C25—P2—C19	104.16 (8)
C15—C16—C17	121.33 (16)	C25—P2—C31	105.01 (8)
C15—C16—Cl3	119.40 (14)	C19—P2—C31	104.90 (7)
C17—C16—Cl3	119.27 (14)	C25—P2—Rh1	119.28 (6)
C16—C17—C18	119.17 (17)	C19—P2—Rh1	110.52 (5)
C16—C17—H17	120.4	C31—P2—Rh1	111.77 (5)
C18—C17—H17	120.4	C37—Rh1—P1	91.58 (5)
C17—C18—C13	120.86 (16)	C37—Rh1—P2	91.37 (5)
C17—C18—H18	119.6	P1—Rh1—P2	175.268 (15)
C13—C18—H18	119.6	C37—Rh1—Cl1	178.21 (5)
C24—C19—C20	119.35 (16)	P1—Rh1—Cl1	86.675 (15)
C24—C19—P2	122.03 (13)	P2—Rh1—Cl1	90.348 (15)
C20—C19—P2	118.57 (13)	O2—C38—C39	120.8 (2)
C21—C20—C19	120.77 (17)	O2—C38—C40	122.3 (2)
C21—C20—H20	119.6	C39—C38—C40	116.8 (2)
C19—C20—H20	119.6	C38—C39—H39A	109.5
C22—C21—C20	118.33 (17)	C38—C39—H39B	109.5
C22—C21—H21	120.8	H39A—C39—H39B	109.5
C20—C21—H21	120.8	C38—C39—H39C	109.5
C23—C22—C21	122.22 (17)	H39A—C39—H39C	109.5
C23—C22—Cl4	118.67 (15)	H39B—C39—H39C	109.5
C21—C22—Cl4	119.08 (15)	C38—C40—H40A	109.5
C22—C23—C24	118.69 (17)	C38—C40—H40B	109.5
C22—C23—H23	120.7	H40A—C40—H40B	109.5
C24—C23—H23	120.7	C38—C40—H40C	109.5
C19—C24—C23	120.54 (17)	H40A—C40—H40C	109.5
C19—C24—H24	119.7	H40B—C40—H40C	109.5
C6—C1—C2—C3	1.3 (2)	C32—C33—C34—Cl6	-176.89 (14)
P1—C1—C2—C3	-175.68 (13)	C33—C34—C35—C36	-2.2 (3)
C1—C2—C3—C4	-0.2 (3)	Cl6—C34—C35—C36	176.83 (15)
C2—C3—C4—C5	-1.7 (3)	C34—C35—C36—C31	0.0 (3)
C2—C3—C4—Cl7	175.40 (14)	C32—C31—C36—C35	2.1 (3)

C3—C4—C5—C6	2.3 (3)	P2—C31—C36—C35	-166.64 (15)
C17—C4—C5—C6	-174.84 (14)	C2—C1—P1—C7	5.09 (17)
C4—C5—C6—C1	-1.0 (3)	C6—C1—P1—C7	-172.00 (12)
C2—C1—C6—C5	-0.7 (3)	C2—C1—P1—C13	-102.85 (15)
P1—C1—C6—C5	176.61 (14)	C6—C1—P1—C13	80.06 (13)
C12—C7—C8—C9	-0.5 (2)	C2—C1—P1—Rh1	129.86 (14)
P1—C7—C8—C9	-175.72 (13)	C6—C1—P1—Rh1	-47.23 (13)
C7—C8—C9—C10	1.4 (3)	C12—C7—P1—C1	123.09 (14)
C8—C9—C10—C11	-1.2 (3)	C8—C7—P1—C1	-61.67 (15)
C8—C9—C10—C12	175.78 (14)	C12—C7—P1—C13	-127.67 (14)
C9—C10—C11—C12	0.1 (3)	C8—C7—P1—C13	47.57 (15)
C12—C10—C11—C12	-176.89 (14)	C12—C7—P1—Rh1	1.59 (15)
C10—C11—C12—C7	0.8 (3)	C8—C7—P1—Rh1	176.83 (12)
C8—C7—C12—C11	-0.6 (3)	C14—C13—P1—C1	-7.18 (16)
P1—C7—C12—C11	174.71 (14)	C18—C13—P1—C1	173.94 (14)
C18—C13—C14—C15	0.2 (3)	C14—C13—P1—C7	-120.32 (15)
P1—C13—C14—C15	-178.63 (14)	C18—C13—P1—C7	60.80 (15)
C13—C14—C15—C16	-1.5 (3)	C14—C13—P1—Rh1	113.46 (14)
C14—C15—C16—C17	1.6 (3)	C18—C13—P1—Rh1	-65.42 (15)
C14—C15—C16—C13	-178.16 (14)	C26—C25—P2—C19	92.47 (15)
C15—C16—C17—C18	-0.4 (3)	C30—C25—P2—C19	-84.32 (16)
C13—C16—C17—C18	179.36 (15)	C26—C25—P2—C31	-157.52 (14)
C16—C17—C18—C13	-0.9 (3)	C30—C25—P2—C31	25.69 (16)
C14—C13—C18—C17	1.0 (3)	C26—C25—P2—Rh1	-31.31 (16)
P1—C13—C18—C17	179.91 (15)	C30—C25—P2—Rh1	151.90 (13)
C24—C19—C20—C21	-1.1 (3)	C24—C19—P2—C25	15.80 (16)
P2—C19—C20—C21	176.43 (14)	C20—C19—P2—C25	-161.65 (14)
C19—C20—C21—C22	-1.5 (3)	C24—C19—P2—C31	-94.30 (15)
C20—C21—C22—C23	3.3 (3)	C20—C19—P2—C31	88.26 (15)
C20—C21—C22—C14	-174.74 (15)	C24—C19—P2—Rh1	145.08 (13)
C21—C22—C23—C24	-2.4 (3)	C20—C19—P2—Rh1	-32.37 (15)
C14—C22—C23—C24	175.62 (15)	C36—C31—P2—C25	-139.07 (14)
C20—C19—C24—C23	2.0 (3)	C32—C31—P2—C25	52.33 (16)
P2—C19—C24—C23	-175.44 (14)	C36—C31—P2—C19	-29.59 (16)
C22—C23—C24—C19	-0.3 (3)	C32—C31—P2—C19	161.81 (14)
C30—C25—C26—C27	-0.9 (3)	C36—C31—P2—Rh1	90.20 (14)
P2—C25—C26—C27	-177.74 (14)	C32—C31—P2—Rh1	-78.39 (15)
C25—C26—C27—C28	-0.1 (3)	C1—P1—Rh1—C37	119.85 (8)
C26—C27—C28—C29	1.2 (3)	C7—P1—Rh1—C37	-118.47 (8)
C26—C27—C28—C15	-178.26 (14)	C13—P1—Rh1—C37	1.69 (8)
C27—C28—C29—C30	-1.2 (3)	C1—P1—Rh1—C11	-60.55 (6)
C15—C28—C29—C30	178.27 (15)	C7—P1—Rh1—C11	61.14 (6)
C28—C29—C30—C25	0.1 (3)	C13—P1—Rh1—C11	-178.70 (6)
C26—C25—C30—C29	0.9 (3)	C25—P2—Rh1—C37	-116.11 (8)
P2—C25—C30—C29	177.70 (14)	C19—P2—Rh1—C37	123.26 (8)
C36—C31—C32—C33	-2.2 (3)	C31—P2—Rh1—C37	6.83 (8)
P2—C31—C32—C33	166.64 (14)	C25—P2—Rh1—C11	64.39 (7)
C31—C32—C33—C34	0.1 (3)	C19—P2—Rh1—C11	-56.23 (6)
C32—C33—C34—C35	2.1 (3)	C31—P2—Rh1—C11	-172.66 (6)

supplementary materials

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C15—H15 \cdots O2 ⁱ	0.95	2.55	3.492 (3)	170
C21—H21 \cdots O2 ⁱⁱ	0.95	2.34	3.267 (3)	165
C24—H24 \cdots C15 ⁱⁱⁱ	0.95	2.80	3.4734 (19)	129
C30—H30 \cdots C14 ^{iv}	0.95	2.76	3.575 (2)	145
C35—H35 \cdots O1 ^v	0.95	2.47	3.384 (2)	161
C40—H40B \cdots C11	0.98	2.79	3.738 (3)	162

Symmetry codes: (i) $x, y+1, z$; (ii) $x+1, y, z$; (iii) $-x+1, -y, -z+1$; (iv) $-x+2, -y, -z+1$; (v) $-x+2, -y+1, -z+1$.

Fig. 1

