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# *trans*-Carbonylchloridobis(tri-*p*-tolylphosphine)rhodium(I) acetone solvate

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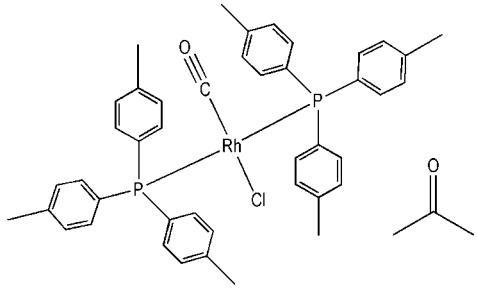
Key indicators: single-crystal X-ray study; $T$ = 173 K; mean $\sigma$(C–C) = 0.006 Å; $R$ factor = 0.048; $wR$ factor = 0.115; data-to-parameter ratio = 20.8.

The title compound, $[RhCl(C_{21}H_{21}P)_2(CO)]\cdot C_3H_6O$, was precipitated in trace yield from a reaction of RhCl(cod)(THP) with P(*p*-tol)$_3$ in a 1:1 acetone-$d_6$/$CD_3OD$ solution under a hydrogen atmosphere [*p*-tol = *p*-tolyl, THP = tris(hydroxymethyl)phosphine, $P(CH_2OH)_3$, and cod = 1,5-cyclooctadiene]. The complex displays a square-planar geometry around the $Rh^I$ atom. The complex molecules and the acetone molecules are linked into a chain along the *a* axis by intermolecular C—H···Cl and C—H···O hydrogen bonds.

## Related literature

For related literature, see: Beck *et al.* (1999, and references therein); Evans *et al.* (1990); Higham *et al.* (2004); Hoye *et al.* (1993); Lorenzini *et al.* (2007*a*,*b*, 2008*a*,*b*); Vallarino (1957).




## Experimental

### *Crystal data*

$[RhCl(C_{21}H_{21}P)_2(CO)]\cdot C_3H_6O$
$M_r$ = 833.14
Triclinic, $P\overline{1}$
$a$ = 10.784 (2) Å
$b$ = 12.859 (3) Å
$c$ = 17.086 (3) Å
$\alpha$ = 70.852 (7)°
$\beta$ = 84.790 (7)°
$\gamma$ = 71.012 (6)°
$V$ = 2116.2 (7) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.58 mm$^{-1}$
$T$ = 173 (2) K
0.25 × 0.10 × 0.07 mm

### *Data collection*

Bruker X8 APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
$T_{min}$ = 0.683, $T_{max}$ = 0.960
31662 measured reflections
9909 independent reflections
6378 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.063

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.115
$S$ = 1.00
9909 reflections
477 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.48 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.44 e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| C43—Rh1 | 1.812 (4) | P2—Rh1 | 2.3283 (9) |
| P1—Rh1 | 2.3449 (9) | Cl1—Rh1 | 2.3822 (9) |
| | | | |
| C43—Rh1—P2 | 91.43 (10) | C43—Rh1—Cl1 | 178.12 (10) |
| C43—Rh1—P1 | 90.55 (10) | P2—Rh1—Cl1 | 86.69 (3) |
| P2—Rh1—P1 | 177.46 (3) | P1—Rh1—Cl1 | 91.33 (3) |

**Table 2**
Hydrogen-bond geometry (Å, °).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C10—H10···O2$^{i}$ | 0.95 | 2.38 | 3.302 (8) | 163 |
| C46—H46*A*···Cl1 | 0.98 | 2.81 | 3.773 (7) | 168 |

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

Data collection: *APEX2* (Bruker, 2006); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); 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: CI2547).

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# supplementary materials

*Acta Cryst.* (2008). E**64**, m464-m465 [ **doi:10.1107/S1600536808003528** ]

# *trans*-Carbonylchloridobis(tri-*p*-tolylphosphine)rhodium(I) acetone solvate

**F. Lorenzini, B. O. Patrick and B. R. James**

## Comment

We have very recently reported the structure of *trans*-RhCl(CO)($PEtPh_2$)$_2$, crystals of which precipitated serendipitously in trace yield from a reaction between $PEtPh_2$ and RhCl(cod)(THP), where cod = 1,5-cyclooctadiene and THP = tris(hydroxymethyl)phosphine, $P(CH_2OH)_3$, in an acetone/MeOH solvent mixture under a hydrogen atmosphere (Lorenzini *et al.*, 2008*b*). Such reaction conditions with a phosphine of general formula P*RR*'$_2$ (*R* = or ≠ *R*') lead to formation of the dihydrido complexes *cis,mer*-Rh(H)$_2$Cl(P*RR*'$_2$)$_3$ (when *R*' = Ph, and *R* = Me or Cy) (Lorenzini *et al.*, 2008*a*) or, if the reaction is carried out under Ar, the phosphine–phosphinite derivatives RhCl(P*RR*'$_2$)[*P*,*P*-*R*'(*R*)$POCH_2P(CH_2OH)_2$] and trace amounts of the *trans*-RhCl(CO)(P*RR*'$_2$)$_2$ species (Lorenzini *et al.*, 2007*b*). The THP plays a critical role in formation of the dihydrides and the carbonyl complexes (Lorenzini *et al.*, 2008*a*); the CO ligand is thought to result from decarbonylation of formaldehyde (Beck *et al.*, 1999), which can be readily formed from transition metal–THP species (Higham *et al.*, 2004; Hoye *et al.*, 1993). A corresponding reaction between the *p*-tolyl phosphine P(*p*-tol)$_3$ and RhCl(cod)(THP) has now similarly led to formation of trace amounts of *trans*-RhCl(CO)[P(*p*-tol)$_3$]$_2$ that was identified by an X-ray structure as an acetone solvated species. The complex has been synthesized previously in high yield from $RhCl_3·3H_2O$ (Evans *et al.*, 1990), while the method first reported 50 years ago used [RhCl(CO)$_2$]$_2$ as the precursor (Vallarino, 1957). Our structure is a further example of the 125 or so of the type with a *trans*-RhCl(CO) moiety associated with two *trans* phosphorus donor atoms (Cambridge Crystallography Data Base).

## Experimental

General. The RhCl(cod)(THP) precursor complex was synthesized by our reported method (Lorenzini *et al.*, 2007*a*); P(*p*-tol)$_3$ (a Strem Chemicals product), and the deuterated solvents (Cambridge Isotope Laboratory) were used as received. The reaction between these reagents was performed under Ar or $H_2$ using standard Schlenk techniques. $^{31}P\{^1H\}$-NMR spectra were measured in acetone-$d_6$/$CD_3OD$ at room temperature (~300 K) on a Bruker AV400 spectrometer, relative to external 85% aq $H_3PO_4$.

*trans*-RhCl(CO)[P(*p*-tol)$_3$]$_2$·$(CH_3)_2CO$. P(*p*-tol)$_3$ (18.3 µl, 0.059 mmol) in acetone-$d_6$ (0.3 ml) was added to a yellow $CD_3OD$ solution (0.3 ml) of RhCl(cod)(THP) (10.2 mg, 0.028 mmol) at room temperature under Ar to give rapid formation of a brown solution. Replacement of the Ar by $H_2$ and subsequent shaking of the vessel resulted in a yellow solution. Over 12 h, a minute quantity of X-ray quality, yellow prism crystals of *trans*-RhCl(CO)[P(*p*-tol)$_3$]$_2$·$(CH_3)_2CO$ deposited from the solution; the $^{31}P\{^1H\}$ spectrum of the solution revealed a complex mixture of species.

### Refinement

H atoms were placed in calculated positions [C—H = 0.95 Å (aromatic) and 0.98 Å (methyl)] and refined using a riding-model approximation, with $U_{iso}(H) = 1.2_{eq}(C)$ and $1.5_{eq}(C_{methyl})$.

### Figures

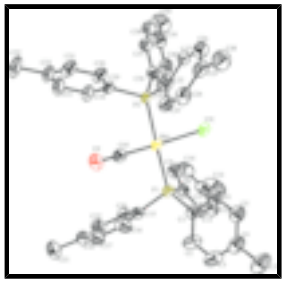

Fig. 1.

### *trans*-Carbonylchloridobis(tri-*p*-tolylphosphine)rhodium(I) acetone solvate

*Crystal data*

| | |
|---|---|
| $[RhCl(C_{21}H_{21}P)_2(CO)]\cdot C_3H_6O$ | $Z = 2$ |
| $M_r = 833.14$ | $F_{000} = 864$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.308$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation<br>$\lambda = 0.71073$ Å |
| $a = 10.784$ (2) Å | Cell parameters from 5165 reflections |
| $b = 12.859$ (3) Å | $\theta = 2.5$–$25.0^o$ |
| $c = 17.086$ (3) Å | $\mu = 0.58$ mm$^{-1}$ |
| $\alpha = 70.852$ (7)º | $T = 173$ (2) K |
| $\beta = 84.790$ (7)º | Prism, yellow |
| $\gamma = 71.012$ (6)º | 0.25 × 0.10 × 0.07 mm |
| $V = 2116.2$ (7) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Bruker X8 APEXII diffractometer | 9909 independent reflections |
| Radiation source: fine-focus sealed tube | 6378 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int} = 0.063$ |
| $T = 173(2)$ K | $\theta_{max} = 27.8^o$ |
| area detector scans | $\theta_{min} = 1.8^o$ |
| Absorption correction: multi-scan (SADABS; Bruker, 2003) | $h = -14\rightarrow14$ |
| $T_{min} = 0.683$, $T_{max} = 0.960$ | $k = -16\rightarrow16$ |
| 31662 measured reflections | $l = -17\rightarrow22$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.047$ | H-atom parameters constrained |
| $wR(F^2) = 0.115$ | $w = 1/[\sigma^2(F_o^2) + (0.045P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.00$ | $(\Delta/\sigma)_{max} = 0.008$ |
| 9909 reflections | $\Delta\rho_{max} = 0.48$ e Å$^{-3}$ |
| 477 parameters | $\Delta\rho_{min} = -0.44$ e Å$^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: none |

*Special details*

**Experimental**. The molecule crystallizes with one molecule of acetone in the asymmetric unit.

**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 $F^2 > 2\sigma(F^2)$ 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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.5990 (3) | 0.2216 (3) | 0.5635 (2) | 0.0401 (8) |
| C2 | 0.5919 (4) | 0.2248 (4) | 0.4811 (2) | 0.0593 (11) |
| H2 | 0.6541 | 0.2482 | 0.4422 | 0.071* |
| C3 | 0.4926 (4) | 0.1932 (4) | 0.4566 (3) | 0.0626 (12) |
| H3 | 0.4885 | 0.1961 | 0.4006 | 0.075* |
| C4 | 0.4011 (4) | 0.1583 (3) | 0.5109 (3) | 0.0500 (10) |
| C5 | 0.4086 (4) | 0.1555 (3) | 0.5919 (2) | 0.0497 (9) |
| H5 | 0.3462 | 0.1316 | 0.6305 | 0.060* |
| C6 | 0.5058 (4) | 0.1871 (3) | 0.6184 (2) | 0.0444 (9) |
| H6 | 0.5082 | 0.1850 | 0.6743 | 0.053* |
| C7 | 0.2945 (4) | 0.1269 (4) | 0.4820 (3) | 0.0721 (13) |
| H7A | 0.2363 | 0.1959 | 0.4424 | 0.108* |
| H7B | 0.2439 | 0.0969 | 0.5297 | 0.108* |
| H7C | 0.3339 | 0.0673 | 0.4552 | 0.108* |
| C8 | 0.8679 (3) | 0.1195 (3) | 0.6269 (2) | 0.0376 (8) |
| C9 | 0.9652 (4) | 0.1031 (3) | 0.6818 (2) | 0.0524 (10) |
| H9 | 0.9593 | 0.1624 | 0.7048 | 0.063* |

| | | | | |
|---|---|---|---|---|
| C10 | 1.0716 (4) | 0.0011 (4) | 0.7039 (2) | 0.0647 (12) |
| H10 | 1.1376 | −0.0074 | 0.7408 | 0.078* |
| C11 | 1.0815 (4) | −0.0885 (3) | 0.6721 (2) | 0.0580 (11) |
| C12 | 0.9877 (4) | −0.0710 (3) | 0.6161 (2) | 0.0568 (11) |
| H12 | 0.9946 | −0.1297 | 0.5923 | 0.068* |
| C13 | 0.8816 (4) | 0.0319 (3) | 0.5932 (2) | 0.0482 (9) |
| H13 | 0.8182 | 0.0417 | 0.5540 | 0.058* |
| C14 | 1.1942 (5) | −0.2021 (4) | 0.6995 (3) | 0.0964 (19) |
| H14A | 1.1901 | −0.2389 | 0.7593 | 0.145* |
| H14B | 1.2779 | −0.1862 | 0.6863 | 0.145* |
| H14C | 1.1870 | −0.2541 | 0.6703 | 0.145* |
| C15 | 0.7857 (3) | 0.3500 (3) | 0.5080 (2) | 0.0384 (8) |
| C16 | 0.6944 (4) | 0.4489 (3) | 0.4564 (2) | 0.0509 (9) |
| H16 | 0.6032 | 0.4585 | 0.4624 | 0.061* |
| C17 | 0.7368 (4) | 0.5328 (3) | 0.3964 (2) | 0.0555 (11) |
| H17 | 0.6734 | 0.5988 | 0.3619 | 0.067* |
| C18 | 0.8699 (4) | 0.5233 (3) | 0.3852 (2) | 0.0502 (10) |
| C19 | 0.9593 (4) | 0.4223 (3) | 0.4345 (2) | 0.0537 (10) |
| H19 | 1.0505 | 0.4112 | 0.4270 | 0.064* |
| C20 | 0.9183 (4) | 0.3373 (3) | 0.4947 (2) | 0.0453 (9) |
| H20 | 0.9820 | 0.2694 | 0.5271 | 0.054* |
| C21 | 0.9137 (5) | 0.6199 (3) | 0.3232 (3) | 0.0717 (13) |
| H21A | 1.0019 | 0.6136 | 0.3385 | 0.107* |
| H21B | 0.8525 | 0.6952 | 0.3234 | 0.107* |
| H21C | 0.9151 | 0.6130 | 0.2676 | 0.107* |
| C22 | 0.4778 (3) | 0.4089 (2) | 0.8594 (2) | 0.0339 (7) |
| C23 | 0.3779 (4) | 0.4611 (3) | 0.7984 (2) | 0.0450 (9) |
| H23 | 0.3992 | 0.4906 | 0.7419 | 0.054* |
| C24 | 0.2485 (4) | 0.4700 (3) | 0.8197 (2) | 0.0499 (9) |
| H24 | 0.1825 | 0.5066 | 0.7774 | 0.060* |
| C25 | 0.2130 (4) | 0.4271 (3) | 0.9009 (3) | 0.0510 (10) |
| C26 | 0.3112 (4) | 0.3759 (3) | 0.9616 (2) | 0.0513 (10) |
| H26 | 0.2888 | 0.3466 | 1.0178 | 0.062* |
| C27 | 0.4431 (4) | 0.3664 (3) | 0.9419 (2) | 0.0434 (9) |
| H27 | 0.5085 | 0.3311 | 0.9845 | 0.052* |
| C28 | 0.0725 (4) | 0.4330 (4) | 0.9218 (3) | 0.0729 (13) |
| H28A | 0.0158 | 0.5136 | 0.9009 | 0.109* |
| H28B | 0.0643 | 0.4036 | 0.9821 | 0.109* |
| H28C | 0.0459 | 0.3855 | 0.8961 | 0.109* |
| C29 | 0.6443 (3) | 0.5526 (2) | 0.80580 (19) | 0.0338 (7) |
| C30 | 0.5354 (4) | 0.6404 (3) | 0.8164 (2) | 0.0430 (9) |
| H30 | 0.4555 | 0.6245 | 0.8342 | 0.052* |
| C31 | 0.5418 (4) | 0.7522 (3) | 0.8012 (2) | 0.0520 (10) |
| H31 | 0.4655 | 0.8117 | 0.8075 | 0.062* |
| C32 | 0.6576 (4) | 0.7778 (3) | 0.7771 (2) | 0.0489 (10) |
| C33 | 0.7671 (4) | 0.6893 (3) | 0.7667 (2) | 0.0543 (10) |
| H33 | 0.8474 | 0.7050 | 0.7498 | 0.065* |
| C34 | 0.7607 (4) | 0.5780 (3) | 0.7807 (2) | 0.0462 (9) |
| H34 | 0.8364 | 0.5189 | 0.7730 | 0.055* |

| | | | | |
|---|---|---|---|---|
| C35 | 0.6639 (5) | 0.8986 (3) | 0.7602 (3) | 0.0728 (14) |
| H35A | 0.6054 | 0.9521 | 0.7133 | 0.109* |
| H35B | 0.7540 | 0.8992 | 0.7469 | 0.109* |
| H35C | 0.6363 | 0.9230 | 0.8094 | 0.109* |
| C36 | 0.7593 (3) | 0.3203 (3) | 0.91027 (19) | 0.0358 (8) |
| C37 | 0.7520 (3) | 0.3574 (3) | 0.9795 (2) | 0.0404 (8) |
| H37 | 0.6827 | 0.4237 | 0.9833 | 0.048* |
| C38 | 0.8462 (4) | 0.2972 (3) | 1.0428 (2) | 0.0495 (9) |
| H38 | 0.8390 | 0.3224 | 1.0899 | 0.059* |
| C39 | 0.9505 (4) | 0.2012 (3) | 1.0386 (2) | 0.0546 (10) |
| C40 | 0.9580 (4) | 0.1654 (3) | 0.9697 (2) | 0.0545 (10) |
| H40 | 1.0287 | 0.1001 | 0.9658 | 0.065* |
| C41 | 0.8637 (3) | 0.2234 (3) | 0.9056 (2) | 0.0445 (9) |
| H41 | 0.8706 | 0.1971 | 0.8590 | 0.053* |
| C42 | 1.0588 (5) | 0.1410 (4) | 1.1040 (3) | 0.0855 (16) |
| H42A | 1.0831 | 0.0569 | 1.1159 | 0.128* |
| H42B | 1.0281 | 0.1605 | 1.1547 | 0.128* |
| H42C | 1.1354 | 0.1665 | 1.0837 | 0.128* |
| C43 | 0.7044 (4) | 0.4689 (3) | 0.6425 (2) | 0.0429 (8) |
| C44 | 0.3191 (5) | 0.0384 (4) | 0.8769 (3) | 0.0736 (13) |
| C45 | 0.3625 (8) | −0.0618 (5) | 0.9469 (4) | 0.163 (3) |
| H45A | 0.3399 | −0.1256 | 0.9391 | 0.245* |
| H45B | 0.3198 | −0.0453 | 0.9966 | 0.245* |
| H45C | 0.4578 | −0.0836 | 0.9535 | 0.245* |
| C46 | 0.3191 (6) | 0.1494 (4) | 0.8789 (4) | 0.122 (2) |
| H46A | 0.3996 | 0.1644 | 0.8540 | 0.183* |
| H46B | 0.3148 | 0.1493 | 0.9365 | 0.183* |
| H46C | 0.2428 | 0.2101 | 0.8476 | 0.183* |
| O1 | 0.7199 (3) | 0.5543 (2) | 0.60038 (16) | 0.0644 (8) |
| O2 | 0.2868 (6) | 0.0305 (5) | 0.8141 (3) | 0.174 (2) |
| P1 | 0.73025 (9) | 0.25561 (7) | 0.60066 (5) | 0.0366 (2) |
| P2 | 0.64329 (8) | 0.40376 (6) | 0.82266 (5) | 0.0322 (2) |
| Cl1 | 0.65594 (10) | 0.15903 (7) | 0.80268 (5) | 0.0504 (2) |
| Rh1 | 0.68398 (3) | 0.33396 (2) | 0.710084 (16) | 0.03563 (9) |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.042 (2) | 0.0352 (18) | 0.042 (2) | −0.0043 (16) | −0.0072 (17) | −0.0173 (16) |
| C2 | 0.058 (3) | 0.082 (3) | 0.049 (2) | −0.023 (2) | 0.005 (2) | −0.036 (2) |
| C3 | 0.062 (3) | 0.083 (3) | 0.056 (3) | −0.021 (2) | −0.010 (2) | −0.039 (2) |
| C4 | 0.045 (2) | 0.042 (2) | 0.064 (3) | −0.0063 (18) | −0.015 (2) | −0.0221 (19) |
| C5 | 0.048 (2) | 0.0373 (19) | 0.060 (3) | −0.0108 (17) | −0.0059 (19) | −0.0121 (18) |
| C6 | 0.052 (2) | 0.0347 (18) | 0.045 (2) | −0.0094 (17) | −0.0073 (18) | −0.0127 (16) |
| C7 | 0.061 (3) | 0.071 (3) | 0.093 (3) | −0.018 (2) | −0.026 (3) | −0.032 (3) |
| C8 | 0.041 (2) | 0.0357 (18) | 0.0349 (19) | −0.0133 (15) | 0.0020 (16) | −0.0092 (15) |
| C9 | 0.059 (3) | 0.050 (2) | 0.050 (2) | −0.014 (2) | −0.009 (2) | −0.0178 (19) |
| C10 | 0.052 (3) | 0.074 (3) | 0.050 (3) | −0.004 (2) | −0.014 (2) | −0.008 (2) |

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|---|---|---|---|---|---|---|
| C11 | 0.058 (3) | 0.051 (2) | 0.042 (2) | 0.002 (2) | 0.007 (2) | −0.0048 (19) |
| C12 | 0.073 (3) | 0.037 (2) | 0.051 (2) | −0.004 (2) | 0.009 (2) | −0.0171 (18) |
| C13 | 0.057 (2) | 0.044 (2) | 0.044 (2) | −0.0122 (19) | −0.0021 (18) | −0.0185 (17) |
| C14 | 0.092 (4) | 0.081 (3) | 0.060 (3) | 0.030 (3) | 0.008 (3) | −0.008 (3) |
| C15 | 0.045 (2) | 0.0369 (18) | 0.0330 (19) | −0.0080 (16) | −0.0008 (16) | −0.0153 (15) |
| C16 | 0.047 (2) | 0.056 (2) | 0.039 (2) | −0.0055 (19) | 0.0022 (18) | −0.0115 (18) |
| C17 | 0.072 (3) | 0.042 (2) | 0.035 (2) | −0.001 (2) | −0.004 (2) | −0.0052 (17) |
| C18 | 0.069 (3) | 0.039 (2) | 0.042 (2) | −0.018 (2) | 0.002 (2) | −0.0110 (17) |
| C19 | 0.052 (2) | 0.047 (2) | 0.058 (3) | −0.0141 (19) | 0.003 (2) | −0.014 (2) |
| C20 | 0.051 (2) | 0.0350 (18) | 0.044 (2) | −0.0099 (17) | −0.0031 (18) | −0.0073 (16) |
| C21 | 0.096 (4) | 0.054 (3) | 0.061 (3) | −0.028 (3) | 0.004 (3) | −0.008 (2) |
| C22 | 0.044 (2) | 0.0234 (15) | 0.0396 (19) | −0.0116 (14) | −0.0024 (16) | −0.0148 (14) |
| C23 | 0.053 (2) | 0.046 (2) | 0.042 (2) | −0.0219 (18) | −0.0039 (18) | −0.0140 (17) |
| C24 | 0.046 (2) | 0.048 (2) | 0.059 (3) | −0.0163 (18) | −0.0058 (19) | −0.0172 (19) |
| C25 | 0.051 (2) | 0.048 (2) | 0.067 (3) | −0.0218 (19) | 0.011 (2) | −0.032 (2) |
| C26 | 0.061 (3) | 0.048 (2) | 0.050 (2) | −0.024 (2) | 0.009 (2) | −0.0179 (19) |
| C27 | 0.058 (2) | 0.0387 (19) | 0.039 (2) | −0.0208 (18) | −0.0018 (18) | −0.0134 (16) |
| C28 | 0.061 (3) | 0.086 (3) | 0.090 (3) | −0.037 (3) | 0.020 (3) | −0.042 (3) |
| C29 | 0.046 (2) | 0.0251 (15) | 0.0323 (18) | −0.0110 (15) | −0.0060 (15) | −0.0097 (13) |
| C30 | 0.049 (2) | 0.0307 (17) | 0.052 (2) | −0.0097 (16) | −0.0042 (18) | −0.0186 (16) |
| C31 | 0.063 (3) | 0.0278 (17) | 0.064 (3) | −0.0041 (18) | −0.014 (2) | −0.0193 (17) |
| C32 | 0.073 (3) | 0.0269 (17) | 0.048 (2) | −0.0181 (19) | −0.022 (2) | −0.0059 (16) |
| C33 | 0.064 (3) | 0.041 (2) | 0.064 (3) | −0.029 (2) | −0.009 (2) | −0.0082 (19) |
| C34 | 0.046 (2) | 0.0339 (18) | 0.061 (2) | −0.0130 (17) | −0.0028 (18) | −0.0166 (17) |
| C35 | 0.103 (4) | 0.0286 (19) | 0.087 (3) | −0.024 (2) | −0.035 (3) | −0.006 (2) |
| C36 | 0.044 (2) | 0.0261 (16) | 0.0368 (19) | −0.0108 (15) | −0.0026 (15) | −0.0084 (14) |
| C37 | 0.047 (2) | 0.0356 (18) | 0.038 (2) | −0.0117 (16) | −0.0026 (17) | −0.0117 (15) |
| C38 | 0.059 (3) | 0.054 (2) | 0.037 (2) | −0.021 (2) | −0.0058 (18) | −0.0114 (18) |
| C39 | 0.054 (3) | 0.050 (2) | 0.053 (2) | −0.017 (2) | −0.011 (2) | −0.0023 (19) |
| C40 | 0.052 (2) | 0.0330 (19) | 0.069 (3) | −0.0040 (18) | −0.006 (2) | −0.0101 (19) |
| C41 | 0.047 (2) | 0.0332 (18) | 0.054 (2) | −0.0105 (17) | −0.0046 (18) | −0.0162 (17) |
| C42 | 0.077 (3) | 0.093 (4) | 0.064 (3) | −0.014 (3) | −0.030 (3) | 0.001 (3) |
| C43 | 0.053 (2) | 0.0378 (19) | 0.040 (2) | −0.0105 (17) | 0.0023 (17) | −0.0192 (17) |
| C44 | 0.086 (4) | 0.065 (3) | 0.079 (3) | −0.020 (3) | −0.011 (3) | −0.035 (3) |
| C45 | 0.255 (10) | 0.069 (4) | 0.099 (5) | −0.003 (5) | 0.012 (5) | 0.008 (4) |
| C46 | 0.131 (5) | 0.068 (4) | 0.176 (6) | −0.039 (4) | 0.006 (5) | −0.044 (4) |
| O1 | 0.098 (2) | 0.0425 (15) | 0.0528 (17) | −0.0303 (16) | 0.0102 (16) | −0.0095 (13) |
| O2 | 0.221 (6) | 0.186 (5) | 0.152 (5) | −0.071 (4) | −0.059 (4) | −0.078 (4) |
| P1 | 0.0443 (5) | 0.0336 (5) | 0.0346 (5) | −0.0106 (4) | −0.0017 (4) | −0.0153 (4) |
| P2 | 0.0424 (5) | 0.0236 (4) | 0.0335 (5) | −0.0105 (4) | −0.0028 (4) | −0.0116 (3) |
| Cl1 | 0.0841 (7) | 0.0353 (4) | 0.0416 (5) | −0.0291 (5) | 0.0002 (5) | −0.0145 (4) |
| Rh1 | 0.04978 (18) | 0.02744 (14) | 0.03398 (16) | −0.01380 (12) | 0.00046 (12) | −0.01354 (11) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—C6 | 1.390 (5) | C25—C26 | 1.389 (5) |
| C1—C2 | 1.403 (5) | C25—C28 | 1.508 (5) |
| C1—P1 | 1.830 (3) | C26—C27 | 1.407 (5) |
| C2—C3 | 1.401 (5) | C26—H26 | 0.95 |

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|---|---|---|---|
| C2—H2 | 0.95 | C27—H27 | 0.95 |
| C3—C4 | 1.373 (5) | C28—H28A | 0.98 |
| C3—H3 | 0.95 | C28—H28B | 0.98 |
| C4—C5 | 1.382 (5) | C28—H28C | 0.98 |
| C4—C7 | 1.505 (5) | C29—C30 | 1.386 (4) |
| C5—C6 | 1.397 (5) | C29—C34 | 1.395 (5) |
| C5—H5 | 0.95 | C29—P2 | 1.843 (3) |
| C6—H6 | 0.95 | C30—C31 | 1.399 (4) |
| C7—H7A | 0.98 | C30—H30 | 0.95 |
| C7—H7B | 0.98 | C31—C32 | 1.387 (5) |
| C7—H7C | 0.98 | C31—H31 | 0.95 |
| C8—C13 | 1.387 (4) | C32—C33 | 1.394 (5) |
| C8—C9 | 1.389 (5) | C32—C35 | 1.506 (4) |
| C8—P1 | 1.840 (3) | C33—C34 | 1.396 (4) |
| C9—C10 | 1.397 (5) | C33—H33 | 0.95 |
| C9—H9 | 0.95 | C34—H34 | 0.95 |
| C10—C11 | 1.399 (6) | C35—H35A | 0.98 |
| C10—H10 | 0.95 | C35—H35B | 0.98 |
| C11—C12 | 1.368 (5) | C35—H35C | 0.98 |
| C11—C14 | 1.526 (5) | C36—C37 | 1.400 (4) |
| C12—C13 | 1.404 (5) | C36—C41 | 1.401 (4) |
| C12—H12 | 0.95 | C36—P2 | 1.836 (3) |
| C13—H13 | 0.95 | C37—C38 | 1.392 (5) |
| C14—H14A | 0.98 | C37—H37 | 0.95 |
| C14—H14B | 0.98 | C38—C39 | 1.391 (5) |
| C14—H14C | 0.98 | C38—H38 | 0.95 |
| C15—C20 | 1.391 (5) | C39—C40 | 1.385 (5) |
| C15—C16 | 1.405 (5) | C39—C42 | 1.515 (5) |
| C15—P1 | 1.837 (3) | C40—C41 | 1.401 (5) |
| C16—C17 | 1.391 (5) | C40—H40 | 0.95 |
| C16—H16 | 0.95 | C41—H41 | 0.95 |
| C17—C18 | 1.400 (5) | C42—H42A | 0.98 |
| C17—H17 | 0.95 | C42—H42B | 0.98 |
| C18—C19 | 1.393 (5) | C42—H42C | 0.98 |
| C18—C21 | 1.523 (5) | C43—O1 | 1.154 (4) |
| C19—C20 | 1.394 (5) | C43—Rh1 | 1.812 (4) |
| C19—H19 | 0.95 | C44—O2 | 1.203 (6) |
| C20—H20 | 0.95 | C44—C45 | 1.417 (6) |
| C21—H21A | 0.98 | C44—C46 | 1.438 (6) |
| C21—H21B | 0.98 | C45—H45A | 0.98 |
| C21—H21C | 0.98 | C45—H45B | 0.98 |
| C22—C27 | 1.398 (4) | C45—H45C | 0.98 |
| C22—C23 | 1.405 (5) | C46—H46A | 0.98 |
| C22—P2 | 1.825 (3) | C46—H46B | 0.98 |
| C23—C24 | 1.387 (5) | C46—H46C | 0.98 |
| C23—H23 | 0.95 | P1—Rh1 | 2.3449 (9) |
| C24—C25 | 1.385 (5) | P2—Rh1 | 2.3283 (9) |
| C24—H24 | 0.95 | Cl1—Rh1 | 2.3822 (9) |
| C6—C1—C2 | 118.3 (3) | C26—C27—H27 | 120.0 |

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| C6—C1—P1 | 119.8 (3) | C25—C28—H28A | 109.5 |
| C2—C1—P1 | 121.9 (3) | C25—C28—H28B | 109.5 |
| C3—C2—C1 | 119.7 (4) | H28A—C28—H28B | 109.5 |
| C3—C2—H2 | 120.2 | C25—C28—H28C | 109.5 |
| C1—C2—H2 | 120.2 | H28A—C28—H28C | 109.5 |
| C4—C3—C2 | 122.1 (4) | H28B—C28—H28C | 109.5 |
| C4—C3—H3 | 119.0 | C30—C29—C34 | 118.4 (3) |
| C2—C3—H3 | 119.0 | C30—C29—P2 | 123.4 (3) |
| C3—C4—C5 | 117.9 (3) | C34—C29—P2 | 118.1 (2) |
| C3—C4—C7 | 120.6 (4) | C29—C30—C31 | 120.7 (3) |
| C5—C4—C7 | 121.5 (4) | C29—C30—H30 | 119.7 |
| C4—C5—C6 | 121.5 (4) | C31—C30—H30 | 119.7 |
| C4—C5—H5 | 119.2 | C32—C31—C30 | 121.2 (3) |
| C6—C5—H5 | 119.2 | C32—C31—H31 | 119.4 |
| C1—C6—C5 | 120.5 (3) | C30—C31—H31 | 119.4 |
| C1—C6—H6 | 119.7 | C31—C32—C33 | 118.0 (3) |
| C5—C6—H6 | 119.7 | C31—C32—C35 | 121.1 (4) |
| C4—C7—H7A | 109.5 | C33—C32—C35 | 120.9 (4) |
| C4—C7—H7B | 109.5 | C32—C33—C34 | 121.0 (4) |
| H7A—C7—H7B | 109.5 | C32—C33—H33 | 119.5 |
| C4—C7—H7C | 109.5 | C34—C33—H33 | 119.5 |
| H7A—C7—H7C | 109.5 | C29—C34—C33 | 120.6 (3) |
| H7B—C7—H7C | 109.5 | C29—C34—H34 | 119.7 |
| C13—C8—C9 | 117.6 (3) | C33—C34—H34 | 119.7 |
| C13—C8—P1 | 123.2 (3) | C32—C35—H35A | 109.5 |
| C9—C8—P1 | 119.2 (3) | C32—C35—H35B | 109.5 |
| C8—C9—C10 | 121.4 (4) | H35A—C35—H35B | 109.5 |
| C8—C9—H9 | 119.3 | C32—C35—H35C | 109.5 |
| C10—C9—H9 | 119.3 | H35A—C35—H35C | 109.5 |
| C9—C10—C11 | 120.4 (4) | H35B—C35—H35C | 109.5 |
| C9—C10—H10 | 119.8 | C37—C36—C41 | 118.8 (3) |
| C11—C10—H10 | 119.8 | C37—C36—P2 | 121.0 (2) |
| C12—C11—C10 | 118.1 (4) | C41—C36—P2 | 120.0 (3) |
| C12—C11—C14 | 121.6 (4) | C38—C37—C36 | 120.1 (3) |
| C10—C11—C14 | 120.4 (4) | C38—C37—H37 | 119.9 |
| C11—C12—C13 | 121.5 (4) | C36—C37—H37 | 119.9 |
| C11—C12—H12 | 119.2 | C39—C38—C37 | 121.5 (3) |
| C13—C12—H12 | 119.2 | C39—C38—H38 | 119.2 |
| C8—C13—C12 | 120.9 (3) | C37—C38—H38 | 119.2 |
| C8—C13—H13 | 119.6 | C40—C39—C38 | 118.2 (3) |
| C12—C13—H13 | 119.6 | C40—C39—C42 | 119.9 (4) |
| C11—C14—H14A | 109.5 | C38—C39—C42 | 121.8 (4) |
| C11—C14—H14B | 109.5 | C39—C40—C41 | 121.5 (3) |
| H14A—C14—H14B | 109.5 | C39—C40—H40 | 119.2 |
| C11—C14—H14C | 109.5 | C41—C40—H40 | 119.2 |
| H14A—C14—H14C | 109.5 | C36—C41—C40 | 119.8 (3) |
| H14B—C14—H14C | 109.5 | C36—C41—H41 | 120.1 |
| C20—C15—C16 | 117.9 (3) | C40—C41—H41 | 120.1 |
| C20—C15—P1 | 121.3 (3) | C39—C42—H42A | 109.5 |

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| C16—C15—P1 | 120.0 (3) | C39—C42—H42B | 109.5 |
| C17—C16—C15 | 120.2 (4) | H42A—C42—H42B | 109.5 |
| C17—C16—H16 | 119.9 | C39—C42—H42C | 109.5 |
| C15—C16—H16 | 119.9 | H42A—C42—H42C | 109.5 |
| C16—C17—C18 | 122.2 (3) | H42B—C42—H42C | 109.5 |
| C16—C17—H17 | 118.9 | O1—C43—Rh1 | 178.5 (3) |
| C18—C17—H17 | 118.9 | O2—C44—C45 | 120.1 (5) |
| C19—C18—C17 | 116.7 (3) | O2—C44—C46 | 119.4 (6) |
| C19—C18—C21 | 121.9 (4) | C45—C44—C46 | 120.5 (5) |
| C17—C18—C21 | 121.3 (4) | C44—C45—H45A | 109.5 |
| C18—C19—C20 | 121.7 (4) | C44—C45—H45B | 109.5 |
| C18—C19—H19 | 119.2 | H45A—C45—H45B | 109.5 |
| C20—C19—H19 | 119.2 | C44—C45—H45C | 109.5 |
| C15—C20—C19 | 121.2 (3) | H45A—C45—H45C | 109.5 |
| C15—C20—H20 | 119.4 | H45B—C45—H45C | 109.5 |
| C19—C20—H20 | 119.4 | C44—C46—H46A | 109.5 |
| C18—C21—H21A | 109.5 | C44—C46—H46B | 109.5 |
| C18—C21—H21B | 109.5 | H46A—C46—H46B | 109.5 |
| H21A—C21—H21B | 109.5 | C44—C46—H46C | 109.5 |
| C18—C21—H21C | 109.5 | H46A—C46—H46C | 109.5 |
| H21A—C21—H21C | 109.5 | H46B—C46—H46C | 109.5 |
| H21B—C21—H21C | 109.5 | C1—P1—C15 | 105.08 (16) |
| C27—C22—C23 | 118.2 (3) | C1—P1—C8 | 104.96 (15) |
| C27—C22—P2 | 125.8 (3) | C15—P1—C8 | 103.74 (15) |
| C23—C22—P2 | 115.9 (3) | C1—P1—Rh1 | 117.85 (12) |
| C24—C23—C22 | 120.6 (3) | C15—P1—Rh1 | 112.37 (10) |
| C24—C23—H23 | 119.7 | C8—P1—Rh1 | 111.61 (11) |
| C22—C23—H23 | 119.7 | C22—P2—C36 | 108.70 (15) |
| C25—C24—C23 | 121.8 (4) | C22—P2—C29 | 102.88 (14) |
| C25—C24—H24 | 119.1 | C36—P2—C29 | 101.69 (14) |
| C23—C24—H24 | 119.1 | C22—P2—Rh1 | 109.37 (10) |
| C24—C25—C26 | 117.9 (4) | C36—P2—Rh1 | 115.03 (11) |
| C24—C25—C28 | 120.6 (4) | C29—P2—Rh1 | 118.15 (11) |
| C26—C25—C28 | 121.5 (4) | C43—Rh1—P2 | 91.43 (10) |
| C25—C26—C27 | 121.6 (4) | C43—Rh1—P1 | 90.55 (10) |
| C25—C26—H26 | 119.2 | P2—Rh1—P1 | 177.46 (3) |
| C27—C26—H26 | 119.2 | C43—Rh1—Cl1 | 178.12 (10) |
| C22—C27—C26 | 119.9 (3) | P2—Rh1—Cl1 | 86.69 (3) |
| C22—C27—H27 | 120.0 | P1—Rh1—Cl1 | 91.33 (3) |
| C6—C1—C2—C3 | −0.2 (6) | C37—C38—C39—C42 | 175.2 (4) |
| P1—C1—C2—C3 | 177.3 (3) | C38—C39—C40—C41 | −0.1 (6) |
| C1—C2—C3—C4 | −0.3 (6) | C42—C39—C40—C41 | −176.1 (4) |
| C2—C3—C4—C5 | 0.4 (6) | C37—C36—C41—C40 | 0.2 (5) |
| C2—C3—C4—C7 | 179.0 (4) | P2—C36—C41—C40 | 174.9 (3) |
| C3—C4—C5—C6 | 0.1 (5) | C39—C40—C41—C36 | 0.4 (6) |
| C7—C4—C5—C6 | −178.6 (3) | C6—C1—P1—C15 | −157.5 (3) |
| C2—C1—C6—C5 | 0.6 (5) | C2—C1—P1—C15 | 25.0 (3) |
| P1—C1—C6—C5 | −176.9 (2) | C6—C1—P1—C8 | 93.4 (3) |
| C4—C5—C6—C1 | −0.5 (5) | C2—C1—P1—C8 | −84.0 (3) |

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|---|---|---|---|
| C13—C8—C9—C10 | −1.4 (6) | C6—C1—P1—Rh1 | −31.5 (3) |
| P1—C8—C9—C10 | 179.8 (3) | C2—C1—P1—Rh1 | 151.1 (3) |
| C8—C9—C10—C11 | −1.2 (6) | C20—C15—P1—C1 | −138.6 (3) |
| C9—C10—C11—C12 | 3.0 (6) | C16—C15—P1—C1 | 52.1 (3) |
| C9—C10—C11—C14 | −177.1 (4) | C20—C15—P1—C8 | −28.7 (3) |
| C10—C11—C12—C13 | −2.3 (6) | C16—C15—P1—C8 | 162.0 (3) |
| C14—C11—C12—C13 | 177.7 (4) | C20—C15—P1—Rh1 | 92.0 (3) |
| C9—C8—C13—C12 | 2.1 (5) | C16—C15—P1—Rh1 | −77.3 (3) |
| P1—C8—C13—C12 | −179.2 (3) | C13—C8—P1—C1 | 23.0 (3) |
| C11—C12—C13—C8 | −0.2 (6) | C9—C8—P1—C1 | −158.4 (3) |
| C20—C15—C16—C17 | −2.6 (5) | C13—C8—P1—C15 | −87.1 (3) |
| P1—C15—C16—C17 | 167.0 (3) | C9—C8—P1—C15 | 91.6 (3) |
| C15—C16—C17—C18 | −0.4 (6) | C13—C8—P1—Rh1 | 151.7 (3) |
| C16—C17—C18—C19 | 3.0 (5) | C9—C8—P1—Rh1 | −29.6 (3) |
| C16—C17—C18—C21 | −175.8 (3) | C27—C22—P2—C36 | 5.7 (3) |
| C17—C18—C19—C20 | −2.7 (5) | C23—C22—P2—C36 | −174.6 (2) |
| C21—C18—C19—C20 | 176.1 (3) | C27—C22—P2—C29 | −101.5 (3) |
| C16—C15—C20—C19 | 2.9 (5) | C23—C22—P2—C29 | 78.1 (3) |
| P1—C15—C20—C19 | −166.6 (3) | C27—C22—P2—Rh1 | 132.1 (2) |
| C18—C19—C20—C15 | −0.2 (5) | C23—C22—P2—Rh1 | −48.3 (2) |
| C27—C22—C23—C24 | 0.0 (5) | C37—C36—P2—C22 | −61.6 (3) |
| P2—C22—C23—C24 | −179.7 (2) | C41—C36—P2—C22 | 123.7 (3) |
| C22—C23—C24—C25 | −0.8 (5) | C37—C36—P2—C29 | 46.4 (3) |
| C23—C24—C25—C26 | 1.1 (5) | C41—C36—P2—C29 | −128.2 (3) |
| C23—C24—C25—C28 | −177.2 (3) | C37—C36—P2—Rh1 | 175.4 (2) |
| C24—C25—C26—C27 | −0.7 (5) | C41—C36—P2—Rh1 | 0.8 (3) |
| C28—C25—C26—C27 | 177.6 (3) | C30—C29—P2—C22 | −3.3 (3) |
| C23—C22—C27—C26 | 0.5 (5) | C34—C29—P2—C22 | 177.0 (3) |
| P2—C22—C27—C26 | −179.9 (2) | C30—C29—P2—C36 | −115.8 (3) |
| C25—C26—C27—C22 | −0.1 (5) | C34—C29—P2—C36 | 64.5 (3) |
| C34—C29—C30—C31 | 0.8 (5) | C30—C29—P2—Rh1 | 117.3 (3) |
| P2—C29—C30—C31 | −178.9 (3) | C34—C29—P2—Rh1 | −62.4 (3) |
| C29—C30—C31—C32 | −1.6 (6) | C22—P2—Rh1—C43 | 117.67 (16) |
| C30—C31—C32—C33 | 1.4 (6) | C36—P2—Rh1—C43 | −119.70 (16) |
| C30—C31—C32—C35 | 179.5 (3) | C29—P2—Rh1—C43 | 0.54 (17) |
| C31—C32—C33—C34 | −0.5 (6) | C22—P2—Rh1—Cl1 | −62.38 (11) |
| C35—C32—C33—C34 | −178.6 (4) | C36—P2—Rh1—Cl1 | 60.25 (12) |
| C30—C29—C34—C33 | 0.1 (5) | C29—P2—Rh1—Cl1 | −179.51 (12) |
| P2—C29—C34—C33 | 179.8 (3) | C1—P1—Rh1—C43 | −114.47 (17) |
| C32—C33—C34—C29 | −0.2 (6) | C15—P1—Rh1—C43 | 7.91 (16) |
| C41—C36—C37—C38 | −1.0 (5) | C8—P1—Rh1—C43 | 123.98 (16) |
| P2—C36—C37—C38 | −175.7 (3) | C1—P1—Rh1—Cl1 | 65.64 (13) |
| C36—C37—C38—C39 | 1.3 (5) | C15—P1—Rh1—Cl1 | −171.98 (12) |
| C37—C38—C39—C40 | −0.7 (6) | C8—P1—Rh1—Cl1 | −55.92 (12) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C10—H10···O2$^{i}$ | 0.95 | 2.38 | 3.302 (8) | 163 |

| | | | | |
|---|---|---|---|---|
| C46—H46A···Cl1 | 0.98 | 2.81 | 3.773 (7) | 168 |

Symmetry codes: (i) *x*+1, *y*, *z*.

**Fig. 1**