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# *cis*-Dichlorido[2,3-dimethyl-3-(4,4,5,5-tetramethyl-1,3,2$\lambda^5$-dioxaphospholan-2-yloxy)butan-2-olato-$\kappa^2O,P$]oxido(triphenylphosphane-$\kappa P$)rhenium(V)

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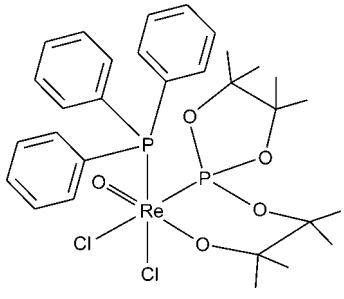
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.005 Å; $R$ factor = 0.026; $wR$ factor = 0.046; data-to-parameter ratio = 23.3.

The title compound, *cis*-[Re($C_{12}H_{24}O_4P$)$Cl_2$O($C_{18}H_{15}P$)], was prepared from the analogous *trans* isomer [Głowiak *et al.* (2000). *Polyhedron*, **19**, 2667–2672] by a *trans–cis* isomerization reaction. The $Re^V$ atom adopts a distorted octahedral coordination geometry. Besides being coordinated by the oxide and the butanolate O atoms, the $Re^V$ atom is coordinated by a pair of chloride ligands and two P atoms in *cis* positions with respect to each other. In the crystal, adjacent molecules are linked by weak C—H···Cl interactions, forming a three-dimensional network.

## Related literature

For related structures and further discussion, see: Głowiak *et al.* (1998, 2000); Rybak *et al.* (2005). For typical bond lengths in coordination complexes, see: Orpen *et al.* (1989). For hydrogen-bond interactions, see: Aullón *et al.* (1998); Desiraju & Steiner (1999); Fábry *et al.* (2004). For details of the temperature control unit used during the data collection, see: Cosier & Glazer (1986). For specifications of the analytical numeric absorption correction, see: Clark & Reid (1995).




## Experimental

### Crystal data

[Re($C_{12}H_{24}O_4P$)$Cl_2$O($C_{18}H_{15}P$)]
$M_r$ = 798.65
Orthorhombic, $P2_12_12_1$
$a$ = 10.963 (3) Å
$b$ = 16.328 (4) Å
$c$ = 17.797 (5) Å
$V$ = 3185.7 (15) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 4.12 mm$^{-1}$
$T$ = 100 K
0.16 × 0.12 × 0.05 mm

### Data collection

Oxford Diffraction Xcalibur PX diffractometer with a CCD detector
Absorption correction: analytical (*CrysAlis RED*; Oxford Diffraction, 2010)
$T_{min}$ = 0.582, $T_{max}$ = 0.808
15943 measured reflections
8614 independent reflections
6813 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.036

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.046
$S$ = 0.87
8614 reflections
369 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.21 e Å$^{-3}$
$\Delta\rho_{min}$ = −1.09 e Å$^{-3}$
Absolute structure: Flack (1983), 3463 Friedel pairs
Flack parameter: −0.015 (4)

**Table 1**
Selected bond lengths (Å).

| | | | |
|---|---|---|---|
| Re1—O1 | 1.698 (2) | Re1—P2 | 2.4883 (12) |
| Re1—O2 | 1.877 (3) | Re1—Cl1 | 2.4461 (10) |
| Re1—P1 | 2.3659 (12) | Re1—Cl2 | 2.4339 (10) |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C31—H31$B$···Cl2$^{i}$ | 0.98 | 2.86 | 3.796 (4) | 161 |
| C42—H42$B$···Cl1$^{ii}$ | 0.98 | 2.87 | 3.830 (4) | 167 |
| C51—H51$B$···Cl1$^{ii}$ | 0.98 | 2.85 | 3.809 (4) | 166 |
| C65—H65···Cl2$^{iii}$ | 0.95 | 2.91 | 3.514 (4) | 123 |

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$; (ii) $-x, y-\frac{1}{2}, -z+\frac{3}{2}$; (iii) $-x+\frac{1}{2}, -y+1, z+\frac{1}{2}$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2010); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2010); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2403).

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# supporting information

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# *cis*-Dichlorido[2,3-dimethyl-3-(4,4,5,5-tetramethyl-1,3,2$\lambda^5$-dioxaphospholan-2-yloxy)butan-2-olato-$\kappa^2O,P$]oxido(triphenylphosphane-$\kappa P$)rhenium(V)

**Anna Skarżyńska, Miłosz Siczek and Andrzej Gniewek**

## S1. Comment

Most of the transition metal derivatives of spirophosphoranes are obtained in ligand substitution reactions, resulting in corresponding metal complexes with $\kappa^2$-*O*,*P* ligand coordination mode (Rybak *et al.*, 2005). In this paper we report the synthesis and crystal structure of the title oxidorhenium(V) complex, obtained starting from an analogous *trans* complex (Głowiak *et al.* 2000). As a result of the *trans*-*cis* isomerization reaction single crystals of the *cis* isomer were obtained.

The coordination environment around the metal center, Re1, is a distorted octahedron with three sets of donor atoms: two O atoms in a *trans* arrangement and two chlorides and both phosphorus located in *cis* positions to each other (Fig. 1). The Re-ligand bond distances (Table 1) are generally similar to those reported for other rhenium complexes, nevertheless some disparities are observed. The distortions of the angles in the coordination sphere of the Re1 atom are significant, for example the O1—Re1—O2 angle of 168.36 (10) ° that differs from the expected value of 180°. The rhenium atom is located 0.06 Å out of the P1/P2/Cl1/Cl2 plane, towards the terminal oxo ligand. The Re1—P2 (phosphane) bond length of 2.4883 (12) Å is within the range 2.42–2.57 Å reported for analogous P$R_3$ derivatives, however the Re1—P1 (phosphite) distance of 2.3659 (12) Å is quite short. This shortening may be explained by the strong $\pi$-acceptor character of the phosphite moiety and is consistent with the Re—P distances observed for other phosphite derivatives (Głowiak *et al.* 1998). The Re1—Cl bond lengths [2.4461 (10) and 2.4339 (10) Å] appear long compared with the expected values of 2.36–2.41 Å (Orpen *et al.*, 1989). This is a result of the high *trans* influence of the phosphorus ligands.

The crystal structure of the title compound is stabilized by a number of weak hydrogen bonds of the C—H···Cl type (Desiraju & Steiner, 1999). Consequently, a three-dimensional network is formed (Table 2). Even though the observed H···Cl distances may first appear to be fairly long compared with the expected values (Aullón *et al.*, 1998), the presence of C—H···Cl hydrogen bonds was confirmed spectroscopically for complexes with H···Cl spacings even above 3 Å (Fábry *et al.*, 2004).

## S2. Experimental

The title compound, *cis*-[$ReOCl_2${$P(OCMe_2CMe_2O)OCMe_2CMe_2O$}$PPh_3$], was prepared from an analogous *trans* isomer, which had been synthesized according to a previously reported procedure (Głowiak *et al.* 2000). The *trans* complex (0.1 g, 0.12 mmol) was dissolved in acetonitrile and refluxed for 6 h. Single crystals of the *cis* isomer suitable for the X-ray analysis were obtained after continuous, slow evaporation of the solvent at ambient temperature. Analysis for [$C_{30}H_{39}Cl_2O_5P_2Re$]: calc. C 45.11, H 4.92; found: C 45.00, H 4.94%. Spectrosopic data for the title compound is given in the archived CIF.

### S3. Refinement

The C-bonded H atoms were positioned geometrically and refined using a riding model: C—H = 0.95 and 0.98 Å for CH and $CH_3$ H atoms, respectively, with $U_{iso}(H) = k \times U_{eq}(C)$, where k = 1.5 for $CH_3$ H atoms, and = 1.2 for other H atoms. In the final difference electron density map the highest residual peak and the deepest hole are located 1.27 and 1.36 Å, respectively, from atom P2.

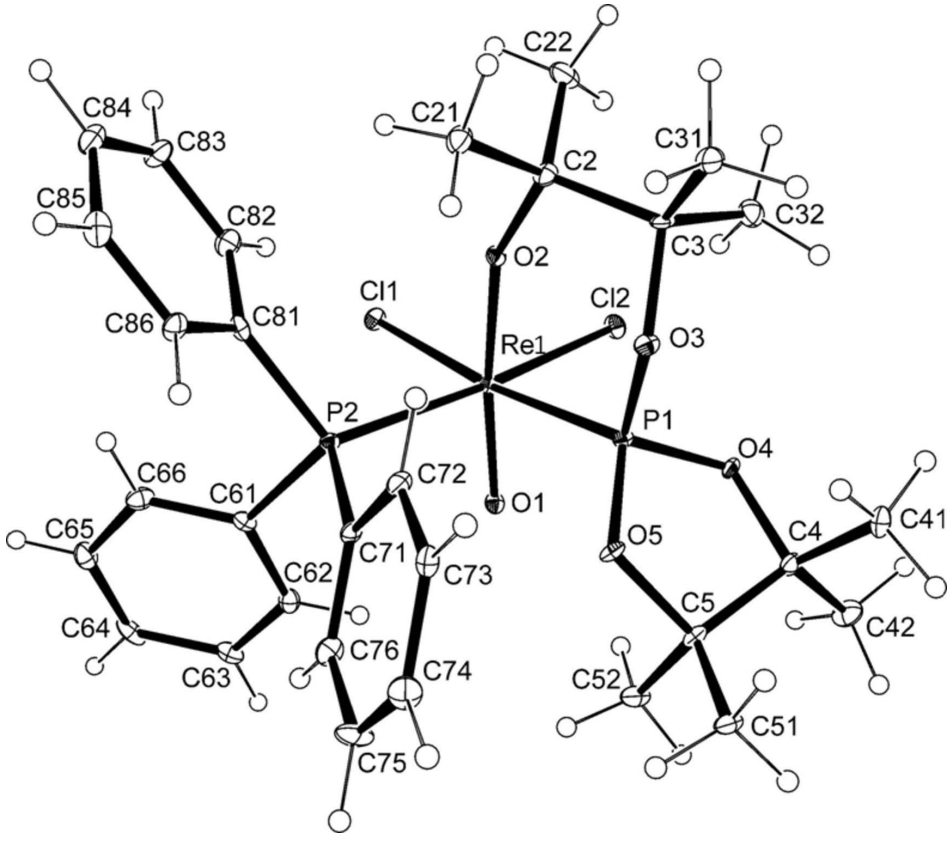


**Figure 1**

The molecular structure of the title compound, with atom numbering. Displacement ellipsoids are drawn at the 30% probability level.

### *cis*-Dichlorido[2,3-dimethyl-3-(4,4,5,5-tetramethyl-1,3,2$\lambda^5$- dioxaphospholan-2-yloxy)butan-2-olato-$\kappa^2O,P$]oxido(triphenylphosphane-$\kappa P$)rhenium(V)

*Crystal data*

$[Re(C_{12}H_{24}O_4P)Cl_2O(C_{18}H_{15}P)]$
$M_r$ = 798.65
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a$ = 10.963 (3) Å
$b$ = 16.328 (4) Å
$c$ = 17.797 (5) Å
$V$ = 3185.7 (15) Å$^3$
$Z$ = 4
$F(000)$ = 1592
$D_x$ = 1.665 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 8908 reflections
$\theta$ = 4.8–30.0°
$\mu$ = 4.12 mm$^{-1}$
$T$ = 100 K
Plate, orange
0.16 × 0.12 × 0.05 mm

*Data collection*

Oxford Diffraction Xcalibur PX diffractometer with a CCD detector
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: analytical (*CrysAlis RED*; Oxford Diffraction, 2010)
$T_{min}$ = 0.582, $T_{max}$ = 0.808
15943 measured reflections
8614 independent reflections
6813 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.036
$\theta_{max}$ = 30.0°, $\theta_{min}$ = 4.8°
$h$ = −14→10
$k$ = −22→15
$l$ = −24→23

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.046
$S$ = 0.87
8614 reflections
369 parameters
0 restraints
Primary atom site location: heavy-atom method
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0111P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 1.21 e Å$^{-3}$
$\Delta\rho_{min}$ = −1.09 e Å$^{-3}$
Absolute structure: Flack (1983), 3463 Friedel pairs
Absolute structure parameter: −0.015 (4)

*Special details*

**Experimental**. Spectrosopic data for the title compound: IR (KBr, cm$^{-1}$): *v*(C—O—P) 926 (*vs*), 941 (*vs*), 1019 (*s*), 1140 (*s*), *v*(Re=O) 955 (*vs*). $^1$H NMR ($CDCl_3$): $\delta$ 0.59, 0.82, 1.18, 1.28, 1.33, 1.43, 1.45, 1.54 (8*H*, s's, $CH_3$), 7.46 (2*H*, m, CH), 7.52 (1*H*, m, CH), 7.76 (2*H*, m, CH). $^{31}$P NMR ($CDCl_3$): $\delta$ -3.67, 87.4 p.p.m..
The crystal was placed in the cold stream of an open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100 K. An analytical numeric absorption correction was carried out with *CrysAlis RED* (Oxford Diffraction, 2010) using a multifaceted crystal model (Clark & Reid, 1995).

**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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Re1 | 0.24960 (2) | 0.319412 (7) | 0.721741 (7) | 0.00859 (3) |
| Cl1 | 0.30747 (8) | 0.46317 (5) | 0.73671 (5) | 0.0139 (2) |
| Cl2 | 0.15281 (9) | 0.36291 (5) | 0.60531 (6) | 0.0153 (2) |
| P1 | 0.20198 (9) | 0.18153 (6) | 0.69328 (6) | 0.01015 (18) |
| P2 | 0.35706 (9) | 0.28754 (5) | 0.84137 (6) | 0.0099 (2) |
| O1 | 0.1159 (2) | 0.32297 (13) | 0.76977 (15) | 0.0121 (5) |
| O2 | 0.3915 (2) | 0.29251 (13) | 0.66814 (15) | 0.0108 (6) |
| O3 | 0.3151 (2) | 0.13616 (13) | 0.65569 (16) | 0.0127 (6) |
| O4 | 0.0825 (2) | 0.17154 (14) | 0.64371 (15) | 0.0120 (6) |
| O5 | 0.1665 (2) | 0.11895 (13) | 0.75739 (15) | 0.0120 (6) |

| | | | | |
|---|---|---|---|---|
| C2 | 0.4648 (4) | 0.2422 (2) | 0.6201 (2) | 0.0138 (9) |
| C21 | 0.5790 (3) | 0.2159 (2) | 0.6628 (3) | 0.0178 (9) |
| H21A | 0.6164 | 0.2639 | 0.6865 | 0.027* |
| H21B | 0.6371 | 0.1911 | 0.6277 | 0.027* |
| H21C | 0.5568 | 0.1759 | 0.7015 | 0.027* |
| C22 | 0.5024 (4) | 0.2971 (2) | 0.5541 (2) | 0.0221 (10) |
| H22A | 0.4295 | 0.3216 | 0.5315 | 0.033* |
| H22B | 0.5453 | 0.2642 | 0.5164 | 0.033* |
| H22C | 0.5565 | 0.3406 | 0.5723 | 0.033* |
| C3 | 0.3877 (3) | 0.1669 (2) | 0.5924 (2) | 0.0119 (8) |
| C31 | 0.4656 (4) | 0.0946 (2) | 0.5698 (2) | 0.0189 (9) |
| H31A | 0.5057 | 0.0719 | 0.6144 | 0.028* |
| H31B | 0.5275 | 0.1124 | 0.5337 | 0.028* |
| H31C | 0.4140 | 0.0525 | 0.5468 | 0.028* |
| C32 | 0.3019 (4) | 0.1889 (2) | 0.5283 (2) | 0.0185 (9) |
| H32A | 0.2460 | 0.1432 | 0.5190 | 0.028* |
| H32B | 0.3496 | 0.1999 | 0.4828 | 0.028* |
| H32C | 0.2550 | 0.2378 | 0.5418 | 0.028* |
| C4 | 0.0126 (4) | 0.0977 (2) | 0.6663 (2) | 0.0137 (8) |
| C41 | 0.0616 (4) | 0.0267 (2) | 0.6204 (3) | 0.0211 (10) |
| H41A | 0.0612 | 0.0415 | 0.5670 | 0.032* |
| H41B | 0.0100 | −0.0215 | 0.6283 | 0.032* |
| H41C | 0.1452 | 0.0144 | 0.6363 | 0.032* |
| C42 | −0.1203 (4) | 0.1148 (2) | 0.6464 (3) | 0.0212 (10) |
| H42A | −0.1444 | 0.1681 | 0.6670 | 0.032* |
| H42B | −0.1722 | 0.0719 | 0.6678 | 0.032* |
| H42C | −0.1297 | 0.1155 | 0.5916 | 0.032* |
| C5 | 0.0380 (3) | 0.0919 (2) | 0.7508 (2) | 0.0123 (8) |
| C51 | 0.0327 (3) | 0.00532 (19) | 0.7833 (3) | 0.0152 (8) |
| H51A | 0.0898 | −0.0300 | 0.7561 | 0.023* |
| H51B | −0.0503 | −0.0163 | 0.7781 | 0.023* |
| H51C | 0.0550 | 0.0068 | 0.8366 | 0.023* |
| C52 | −0.0384 (4) | 0.1492 (2) | 0.7990 (2) | 0.0182 (9) |
| H52A | −0.0049 | 0.1509 | 0.8500 | 0.027* |
| H52B | −0.1227 | 0.1293 | 0.8007 | 0.027* |
| H52C | −0.0368 | 0.2043 | 0.7772 | 0.027* |
| C61 | 0.3018 (5) | 0.3519 (3) | 0.9191 (3) | 0.0120 (10) |
| C62 | 0.1749 (4) | 0.3585 (3) | 0.9323 (3) | 0.0151 (11) |
| H62 | 0.1184 | 0.3309 | 0.9007 | 0.018* |
| C63 | 0.1342 (4) | 0.4053 (2) | 0.9912 (3) | 0.0158 (10) |
| H63 | 0.0492 | 0.4087 | 1.0011 | 0.019* |
| C64 | 0.2152 (4) | 0.4477 (2) | 1.0365 (3) | 0.0204 (13) |
| H64 | 0.1863 | 0.4802 | 1.0770 | 0.024* |
| C65 | 0.3385 (4) | 0.4421 (2) | 1.0219 (3) | 0.0188 (10) |
| H65 | 0.3943 | 0.4717 | 1.0524 | 0.023* |
| C66 | 0.3823 (4) | 0.3943 (2) | 0.9639 (3) | 0.0172 (11) |
| H66 | 0.4676 | 0.3907 | 0.9550 | 0.021* |
| C71 | 0.3430 (3) | 0.1828 (2) | 0.8771 (2) | 0.0112 (7) |

| | | | | |
|---|---|---|---|---|
| C72 | 0.4035 (3) | 0.1196 (2) | 0.8402 (2) | 0.0146 (8) |
| H72 | 0.4541 | 0.1316 | 0.7983 | 0.018* |
| C73 | 0.3906 (4) | 0.0390 (2) | 0.8641 (2) | 0.0140 (8) |
| H73 | 0.4335 | −0.0037 | 0.8394 | 0.017* |
| C74 | 0.3157 (4) | 0.0214 (2) | 0.9236 (3) | 0.0216 (10) |
| H74 | 0.3058 | −0.0338 | 0.9392 | 0.026* |
| C75 | 0.2541 (6) | 0.08333 (18) | 0.9614 (2) | 0.0217 (8) |
| H75 | 0.2025 | 0.0707 | 1.0027 | 0.026* |
| C76 | 0.2690 (4) | 0.16424 (19) | 0.9379 (2) | 0.0168 (10) |
| H76 | 0.2279 | 0.2070 | 0.9637 | 0.020* |
| C81 | 0.5210 (3) | 0.3057 (2) | 0.8386 (2) | 0.0130 (8) |
| C82 | 0.5652 (4) | 0.3742 (2) | 0.8016 (2) | 0.0176 (9) |
| H82 | 0.5099 | 0.4106 | 0.7776 | 0.021* |
| C83 | 0.6886 (4) | 0.3902 (2) | 0.7994 (2) | 0.0198 (10) |
| H83 | 0.7176 | 0.4376 | 0.7740 | 0.024* |
| C84 | 0.7713 (4) | 0.33728 (19) | 0.8340 (2) | 0.0187 (10) |
| H84 | 0.8564 | 0.3475 | 0.8312 | 0.022* |
| C85 | 0.7275 (4) | 0.2695 (2) | 0.8726 (2) | 0.0186 (11) |
| H85 | 0.7827 | 0.2335 | 0.8972 | 0.022* |
| C86 | 0.6027 (4) | 0.2543 (2) | 0.8753 (2) | 0.0147 (9) |
| H86 | 0.5730 | 0.2084 | 0.9026 | 0.018* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Re1 | 0.00898 (5) | 0.00806 (5) | 0.00874 (5) | 0.00034 (11) | 0.00038 (11) | 0.00042 (6) |
| Cl1 | 0.0147 (4) | 0.0095 (4) | 0.0175 (6) | −0.0004 (4) | −0.0008 (4) | −0.0002 (4) |
| Cl2 | 0.0188 (5) | 0.0149 (4) | 0.0123 (5) | 0.0020 (4) | −0.0035 (4) | 0.0022 (4) |
| P1 | 0.0096 (4) | 0.0098 (4) | 0.0111 (5) | 0.0000 (4) | 0.0007 (4) | 0.0001 (4) |
| P2 | 0.0103 (5) | 0.0090 (4) | 0.0103 (5) | −0.0007 (4) | 0.0009 (4) | 0.0005 (4) |
| O1 | 0.0119 (12) | 0.0116 (11) | 0.0128 (14) | 0.0021 (11) | −0.0004 (11) | −0.0025 (12) |
| O2 | 0.0115 (13) | 0.0099 (11) | 0.0109 (15) | 0.0009 (10) | 0.0006 (12) | −0.0020 (11) |
| O3 | 0.0121 (14) | 0.0088 (11) | 0.0172 (16) | 0.0009 (11) | 0.0043 (12) | −0.0028 (11) |
| O4 | 0.0118 (13) | 0.0113 (12) | 0.0127 (15) | −0.0056 (11) | −0.0016 (11) | −0.0002 (11) |
| O5 | 0.0073 (13) | 0.0110 (12) | 0.0177 (16) | −0.0029 (10) | 0.0002 (11) | 0.0031 (10) |
| C2 | 0.0093 (19) | 0.0145 (19) | 0.018 (2) | 0.0020 (16) | 0.0008 (16) | −0.0021 (16) |
| C21 | 0.013 (2) | 0.0197 (19) | 0.021 (2) | 0.0000 (17) | −0.0012 (18) | −0.0044 (18) |
| C22 | 0.032 (3) | 0.019 (2) | 0.015 (2) | −0.0042 (19) | 0.008 (2) | −0.0013 (17) |
| C3 | 0.0088 (18) | 0.012 (2) | 0.014 (2) | −0.0009 (15) | 0.0063 (15) | −0.0008 (16) |
| C31 | 0.017 (2) | 0.019 (2) | 0.021 (3) | −0.0040 (18) | 0.0053 (19) | −0.0056 (18) |
| C32 | 0.0186 (19) | 0.0193 (19) | 0.018 (2) | 0.0014 (19) | 0.0002 (17) | −0.0032 (18) |
| C4 | 0.015 (2) | 0.0105 (17) | 0.015 (2) | −0.0044 (16) | −0.0013 (18) | 0.0012 (17) |
| C41 | 0.025 (2) | 0.017 (2) | 0.021 (3) | −0.0075 (19) | 0.000 (2) | −0.0085 (18) |
| C42 | 0.011 (2) | 0.027 (2) | 0.025 (3) | −0.0013 (18) | −0.0033 (19) | 0.0050 (19) |
| C5 | 0.0083 (18) | 0.0106 (17) | 0.018 (2) | −0.0027 (15) | −0.0002 (16) | 0.0013 (16) |
| C51 | 0.0129 (18) | 0.0144 (17) | 0.018 (2) | −0.0037 (15) | 0.0045 (19) | 0.0018 (18) |
| C52 | 0.020 (2) | 0.0138 (18) | 0.020 (3) | −0.0009 (17) | 0.0081 (18) | 0.0010 (16) |
| C61 | 0.017 (2) | 0.0074 (19) | 0.011 (2) | 0.0015 (17) | 0.0019 (19) | 0.0020 (16) |

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|---|---|---|---|---|---|---|
| C62 | 0.016 (2) | 0.014 (2) | 0.015 (3) | 0.001 (2) | −0.003 (2) | 0.0004 (19) |
| C63 | 0.019 (2) | 0.015 (2) | 0.014 (3) | 0.0072 (19) | 0.003 (2) | 0.0048 (19) |
| C64 | 0.042 (4) | 0.0092 (17) | 0.010 (2) | 0.0057 (18) | 0.0033 (19) | −0.0015 (15) |
| C65 | 0.028 (3) | 0.013 (2) | 0.016 (3) | −0.003 (2) | −0.003 (2) | −0.0015 (18) |
| C66 | 0.017 (2) | 0.013 (2) | 0.022 (3) | −0.0013 (18) | 0.004 (2) | −0.0001 (19) |
| C71 | 0.0128 (18) | 0.0075 (15) | 0.013 (2) | −0.0026 (17) | −0.0013 (15) | 0.0009 (17) |
| C72 | 0.0113 (19) | 0.0163 (19) | 0.016 (2) | −0.0032 (16) | −0.0008 (17) | 0.0007 (17) |
| C73 | 0.016 (2) | 0.0116 (17) | 0.015 (2) | −0.0005 (17) | −0.0046 (17) | −0.0018 (16) |
| C74 | 0.030 (3) | 0.013 (2) | 0.021 (3) | −0.0036 (18) | −0.004 (2) | 0.0073 (17) |
| C75 | 0.030 (2) | 0.0160 (15) | 0.019 (2) | −0.002 (3) | 0.008 (3) | 0.0074 (14) |
| C76 | 0.017 (3) | 0.0129 (17) | 0.021 (2) | 0.0001 (16) | −0.0001 (17) | −0.0020 (13) |
| C81 | 0.0172 (19) | 0.0130 (19) | 0.009 (2) | 0.0002 (16) | −0.0014 (16) | −0.0031 (16) |
| C82 | 0.016 (2) | 0.0163 (19) | 0.021 (2) | 0.0007 (17) | −0.0018 (17) | −0.0005 (17) |
| C83 | 0.014 (2) | 0.0195 (19) | 0.026 (3) | −0.0048 (17) | −0.0013 (18) | 0.0036 (17) |
| C84 | 0.012 (3) | 0.0197 (18) | 0.025 (2) | −0.0008 (15) | −0.0019 (17) | −0.0050 (15) |
| C85 | 0.019 (3) | 0.0146 (16) | 0.022 (2) | 0.0078 (17) | −0.0052 (17) | −0.0044 (15) |
| C86 | 0.017 (2) | 0.0096 (17) | 0.018 (2) | −0.0048 (16) | −0.0029 (17) | 0.0017 (16) |

*Geometric parameters (Å, º)*

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| Re1—O1 | 1.698 (2) | C5—C52 | 1.520 (5) |
| Re1—O2 | 1.877 (3) | C5—C51 | 1.529 (5) |
| Re1—P1 | 2.3659 (12) | C51—H51A | 0.9800 |
| Re1—P2 | 2.4883 (12) | C51—H51B | 0.9800 |
| Re1—Cl1 | 2.4461 (10) | C51—H51C | 0.9800 |
| Re1—Cl2 | 2.4339 (10) | C52—H52A | 0.9800 |
| P1—O3 | 1.592 (3) | C52—H52B | 0.9800 |
| P1—O4 | 1.588 (3) | C52—H52C | 0.9800 |
| P1—O5 | 1.580 (3) | C61—C66 | 1.377 (6) |
| P2—C61 | 1.840 (4) | C61—C62 | 1.415 (5) |
| P2—C71 | 1.831 (4) | C62—C63 | 1.372 (6) |
| P2—C81 | 1.822 (4) | C62—H62 | 0.9500 |
| O2—C2 | 1.432 (4) | C63—C64 | 1.384 (6) |
| O3—C3 | 1.468 (4) | C63—H63 | 0.9500 |
| O4—C4 | 1.484 (4) | C64—C65 | 1.380 (6) |
| O5—C5 | 1.481 (4) | C64—H64 | 0.9500 |
| C2—C21 | 1.526 (5) | C65—C66 | 1.380 (6) |
| C2—C22 | 1.534 (5) | C65—H65 | 0.9500 |
| C2—C3 | 1.571 (5) | C66—H66 | 0.9500 |
| C21—H21A | 0.9800 | C71—C76 | 1.386 (5) |
| C21—H21B | 0.9800 | C71—C72 | 1.391 (5) |
| C21—H21C | 0.9800 | C72—C73 | 1.390 (5) |
| C22—H22A | 0.9800 | C72—H72 | 0.9500 |
| C22—H22B | 0.9800 | C73—C74 | 1.369 (6) |
| C22—H22C | 0.9800 | C73—H73 | 0.9500 |
| C3—C31 | 1.511 (5) | C74—C75 | 1.390 (6) |
| C3—C32 | 1.520 (5) | C74—H74 | 0.9500 |
| C31—H31A | 0.9800 | C75—C76 | 1.395 (4) |

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| C31—H31B | 0.9800 | C75—H75 | 0.9500 |
| C31—H31C | 0.9800 | C76—H76 | 0.9500 |
| C32—H32A | 0.9800 | C81—C82 | 1.386 (5) |
| C32—H32B | 0.9800 | C81—C86 | 1.390 (5) |
| C32—H32C | 0.9800 | C82—C83 | 1.378 (5) |
| C4—C41 | 1.515 (5) | C82—H82 | 0.9500 |
| C4—C42 | 1.525 (5) | C83—C84 | 1.396 (5) |
| C4—C5 | 1.534 (6) | C83—H83 | 0.9500 |
| C41—H41A | 0.9800 | C84—C85 | 1.389 (5) |
| C41—H41B | 0.9800 | C84—H84 | 0.9500 |
| C41—H41C | 0.9800 | C85—C86 | 1.391 (6) |
| C42—H42A | 0.9800 | C85—H85 | 0.9500 |
| C42—H42B | 0.9800 | C86—H86 | 0.9500 |
| C42—H42C | 0.9800 | | |

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| O1—Re1—O2 | 168.36 (10) | C4—C42—H42A | 109.5 |
| O1—Re1—P1 | 87.13 (8) | C4—C42—H42B | 109.5 |
| O2—Re1—P1 | 81.45 (8) | H42A—C42—H42B | 109.5 |
| O1—Re1—P2 | 89.15 (9) | C4—C42—H42C | 109.5 |
| O2—Re1—P2 | 89.63 (9) | H42A—C42—H42C | 109.5 |
| O1—Re1—Cl2 | 92.42 (9) | H42B—C42—H42C | 109.5 |
| O2—Re1—Cl2 | 89.82 (9) | O5—C5—C52 | 107.3 (3) |
| P1—Re1—Cl2 | 89.96 (3) | O5—C5—C51 | 106.4 (3) |
| O1—Re1—Cl1 | 97.83 (8) | C52—C5—C51 | 109.6 (3) |
| O2—Re1—Cl1 | 93.73 (8) | O5—C5—C4 | 103.4 (3) |
| P1—Re1—Cl1 | 173.56 (3) | C52—C5—C4 | 114.5 (3) |
| P2—Re1—Cl2 | 174.79 (3) | C51—C5—C4 | 114.9 (3) |
| Cl1—Re1—Cl2 | 85.74 (3) | C5—C51—H51A | 109.5 |
| P1—Re1—P2 | 95.09 (3) | C5—C51—H51B | 109.5 |
| Cl1—Re1—P2 | 89.12 (3) | H51A—C51—H51B | 109.5 |
| O5—P1—O4 | 97.57 (14) | C5—C51—H51C | 109.5 |
| O5—P1—O3 | 101.19 (13) | H51A—C51—H51C | 109.5 |
| O4—P1—O3 | 111.19 (15) | H51B—C51—H51C | 109.5 |
| O5—P1—Re1 | 121.00 (10) | C5—C52—H52A | 109.5 |
| O4—P1—Re1 | 113.50 (10) | C5—C52—H52B | 109.5 |
| O3—P1—Re1 | 111.16 (9) | H52A—C52—H52B | 109.5 |
| C81—P2—C71 | 104.11 (17) | C5—C52—H52C | 109.5 |
| C81—P2—C61 | 104.6 (2) | H52A—C52—H52C | 109.5 |
| C71—P2—C61 | 104.15 (19) | H52B—C52—H52C | 109.5 |
| C81—P2—Re1 | 114.21 (13) | C66—C61—C62 | 119.7 (5) |
| C71—P2—Re1 | 116.91 (12) | C66—C61—P2 | 120.8 (4) |
| C61—P2—Re1 | 111.59 (15) | C62—C61—P2 | 119.5 (4) |
| C2—O2—Re1 | 154.5 (2) | C63—C62—C61 | 119.3 (5) |
| C3—O3—P1 | 125.9 (2) | C63—C62—H62 | 120.4 |
| C4—O4—P1 | 111.0 (2) | C61—C62—H62 | 120.4 |
| C5—O5—P1 | 111.7 (2) | C62—C63—C64 | 121.0 (4) |
| O2—C2—C21 | 109.0 (3) | C62—C63—H63 | 119.5 |
| O2—C2—C22 | 105.8 (3) | C64—C63—H63 | 119.5 |

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| C21—C2—C22 | 108.9 (3) | C65—C64—C63 | 119.0 (5) |
| O2—C2—C3 | 109.6 (3) | C65—C64—H64 | 120.5 |
| C21—C2—C3 | 112.2 (3) | C63—C64—H64 | 120.5 |
| C22—C2—C3 | 111.1 (3) | C64—C65—C66 | 121.3 (5) |
| C2—C21—H21A | 109.5 | C64—C65—H65 | 119.4 |
| C2—C21—H21B | 109.5 | C66—C65—H65 | 119.4 |
| H21A—C21—H21B | 109.5 | C61—C66—C65 | 119.7 (5) |
| C2—C21—H21C | 109.5 | C61—C66—H66 | 120.2 |
| H21A—C21—H21C | 109.5 | C65—C66—H66 | 120.2 |
| H21B—C21—H21C | 109.5 | C76—C71—C72 | 119.0 (3) |
| C2—C22—H22A | 109.5 | C76—C71—P2 | 121.6 (3) |
| C2—C22—H22B | 109.5 | C72—C71—P2 | 119.3 (3) |
| H22A—C22—H22B | 109.5 | C73—C72—C71 | 120.6 (4) |
| C2—C22—H22C | 109.5 | C73—C72—H72 | 119.7 |
| H22A—C22—H22C | 109.5 | C71—C72—H72 | 119.7 |
| H22B—C22—H22C | 109.5 | C74—C73—C72 | 119.8 (4) |
| O3—C3—C31 | 104.1 (3) | C74—C73—H73 | 120.1 |
| O3—C3—C32 | 108.7 (3) | C72—C73—H73 | 120.1 |
| C31—C3—C32 | 109.6 (3) | C73—C74—C75 | 120.8 (4) |
| O3—C3—C2 | 108.5 (3) | C73—C74—H74 | 119.6 |
| C31—C3—C2 | 113.0 (3) | C75—C74—H74 | 119.6 |
| C32—C3—C2 | 112.6 (3) | C74—C75—C76 | 119.1 (4) |
| C3—C31—H31A | 109.5 | C74—C75—H75 | 120.4 |
| C3—C31—H31B | 109.5 | C76—C75—H75 | 120.4 |
| H31A—C31—H31B | 109.5 | C71—C76—C75 | 120.7 (4) |
| C3—C31—H31C | 109.5 | C71—C76—H76 | 119.7 |
| H31A—C31—H31C | 109.5 | C75—C76—H76 | 119.7 |
| H31B—C31—H31C | 109.5 | C82—C81—C86 | 119.1 (3) |
| C3—C32—H32A | 109.5 | C82—C81—P2 | 119.2 (3) |
| C3—C32—H32B | 109.5 | C86—C81—P2 | 121.6 (3) |
| H32A—C32—H32B | 109.5 | C83—C82—C81 | 120.7 (4) |
| C3—C32—H32C | 109.5 | C83—C82—H82 | 119.7 |
| H32A—C32—H32C | 109.5 | C81—C82—H82 | 119.7 |
| H32B—C32—H32C | 109.5 | C82—C83—C84 | 120.5 (4) |
| O4—C4—C41 | 107.0 (3) | C82—C83—H83 | 119.8 |
| O4—C4—C42 | 106.3 (3) | C84—C83—H83 | 119.8 |
| C41—C4—C42 | 110.7 (3) | C85—C84—C83 | 119.2 (4) |
| O4—C4—C5 | 102.8 (3) | C85—C84—H84 | 120.4 |
| C41—C4—C5 | 114.6 (3) | C83—C84—H84 | 120.4 |
| C42—C4—C5 | 114.4 (4) | C84—C85—C86 | 119.9 (4) |
| C4—C41—H41A | 109.5 | C84—C85—H85 | 120.0 |
| C4—C41—H41B | 109.5 | C86—C85—H85 | 120.0 |
| H41A—C41—H41B | 109.5 | C81—C86—C85 | 120.6 (4) |
| C4—C41—H41C | 109.5 | C81—C86—H86 | 119.7 |
| H41A—C41—H41C | 109.5 | C85—C86—H86 | 119.7 |
| H41B—C41—H41C | 109.5 | | |
| | | | |
| O1—RE1—P1—O5 | −51.92 (14) | P1—O4—C4—C5 | 32.4 (3) |

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| O2—RE1—P1—O5 | 125.82 (14) | P1—O5—C5—C52 | −92.2 (3) |
| Cl2—RE1—P1—O5 | −144.35 (12) | P1—O5—C5—C51 | 150.6 (2) |
| P2—RE1—P1—O5 | 36.96 (12) | P1—O5—C5—C4 | 29.1 (3) |
| O1—RE1—P1—O4 | 63.47 (14) | O4—C4—C5—O5 | −36.4 (3) |
| O2—RE1—P1—O4 | −118.78 (14) | C41—C4—C5—O5 | 79.3 (4) |
| Cl2—RE1—P1—O4 | −28.96 (12) | C42—C4—C5—O5 | −151.2 (3) |
| P2—RE1—P1—O4 | 152.35 (12) | O4—C4—C5—C52 | 79.9 (4) |
| O1—RE1—P1—O3 | −170.31 (15) | C41—C4—C5—C52 | −164.3 (3) |
| O2—RE1—P1—O3 | 7.44 (14) | C42—C4—C5—C52 | −34.9 (4) |
| Cl2—RE1—P1—O3 | 97.26 (12) | O4—C4—C5—C51 | −151.9 (3) |
| P2—RE1—P1—O3 | −81.42 (12) | C41—C4—C5—C51 | −36.2 (5) |
| O1—RE1—P2—C81 | −161.66 (14) | C42—C4—C5—C51 | 93.2 (4) |
| O2—RE1—P2—C81 | 29.91 (14) | C81—P2—C61—C66 | −5.3 (4) |
| P1—RE1—P2—C81 | 111.30 (12) | C71—P2—C61—C66 | 103.7 (4) |
| Cl1—RE1—P2—C81 | −63.82 (12) | RE1—P2—C61—C66 | −129.3 (3) |
| O1—RE1—P2—C71 | 76.47 (16) | C81—P2—C61—C62 | 173.9 (4) |
| O2—RE1—P2—C71 | −91.95 (15) | C71—P2—C61—C62 | −77.1 (5) |
| P1—RE1—P2—C71 | −10.57 (14) | RE1—P2—C61—C62 | 49.9 (5) |
| Cl1—RE1—P2—C71 | 174.32 (14) | C66—C61—C62—C63 | −2.0 (8) |
| O1—RE1—P2—C61 | −43.24 (18) | P2—C61—C62—C63 | 178.8 (3) |
| O2—RE1—P2—C61 | 148.34 (17) | C61—C62—C63—C64 | 1.8 (7) |
| P1—RE1—P2—C61 | −130.28 (16) | C62—C63—C64—C65 | −0.3 (7) |
| Cl1—RE1—P2—C61 | 54.60 (16) | C63—C64—C65—C66 | −0.9 (7) |
| O1—RE1—O2—C2 | 23.7 (10) | C62—C61—C66—C65 | 0.8 (7) |
| P1—RE1—O2—C2 | 12.4 (6) | P2—C61—C66—C65 | −180.0 (3) |
| Cl2—RE1—O2—C2 | −77.5 (6) | C64—C65—C66—C61 | 0.6 (7) |
| Cl1—RE1—O2—C2 | −163.3 (6) | C81—P2—C71—C76 | 127.8 (3) |
| P2—RE1—O2—C2 | 107.6 (6) | C61—P2—C71—C76 | 18.4 (4) |
| O5—P1—O3—C3 | −179.1 (3) | RE1—P2—C71—C76 | −105.2 (3) |
| O4—P1—O3—C3 | 78.1 (3) | C81—P2—C71—C72 | −55.3 (3) |
| RE1—P1—O3—C3 | −49.4 (3) | C61—P2—C71—C72 | −164.7 (3) |
| O5—P1—O4—C4 | −14.6 (3) | RE1—P2—C71—C72 | 71.7 (3) |
| O3—P1—O4—C4 | 90.5 (2) | C76—C71—C72—C73 | −0.4 (6) |
| RE1—P1—O4—C4 | −143.3 (2) | P2—C71—C72—C73 | −177.4 (3) |
| O4—P1—O5—C5 | −9.6 (2) | C71—C72—C73—C74 | 1.3 (6) |
| O3—P1—O5—C5 | −123.1 (2) | C72—C73—C74—C75 | −1.2 (7) |
| RE1—P1—O5—C5 | 113.7 (2) | C73—C74—C75—C76 | 0.2 (8) |
| RE1—O2—C2—C21 | −119.4 (5) | C72—C71—C76—C75 | −0.6 (7) |
| RE1—O2—C2—C22 | 123.7 (5) | P2—C71—C76—C75 | 176.2 (4) |
| RE1—O2—C2—C3 | 3.8 (7) | C74—C75—C76—C71 | 0.7 (8) |
| P1—O3—C3—C31 | −169.5 (2) | C71—P2—C81—C82 | 168.6 (3) |
| P1—O3—C3—C32 | −52.8 (4) | C61—P2—C81—C82 | −82.3 (4) |
| P1—O3—C3—C2 | 70.0 (4) | RE1—P2—C81—C82 | 40.0 (3) |
| O2—C2—C3—O3 | −41.2 (4) | C71—P2—C81—C86 | −14.1 (4) |
| C21—C2—C3—O3 | 80.0 (4) | C61—P2—C81—C86 | 94.9 (3) |
| C22—C2—C3—O3 | −157.8 (3) | RE1—P2—C81—C86 | −142.8 (3) |
| O2—C2—C3—C31 | −156.1 (3) | C86—C81—C82—C83 | 1.8 (6) |
| C21—C2—C3—C31 | −34.9 (5) | P2—C81—C82—C83 | 179.2 (3) |

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| C22—C2—C3—C31 | 87.4 (4) | C81—C82—C83—C84 | 0.3 (7) |
| O2—C2—C3—C32 | 79.2 (4) | C82—C83—C84—C85 | −1.8 (6) |
| C21—C2—C3—C32 | −159.6 (3) | C83—C84—C85—C86 | 1.1 (6) |
| C22—C2—C3—C32 | −37.4 (4) | C82—C81—C86—C85 | −2.5 (6) |
| P1—O4—C4—C41 | −88.7 (3) | P2—C81—C86—C85 | −179.8 (3) |
| P1—O4—C4—C42 | 152.9 (3) | C84—C85—C86—C81 | 1.1 (6) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C31—H31*B*···Cl2$^{i}$ | 0.98 | 2.86 | 3.796 (4) | 161 |
| C42—H42*B*···Cl1$^{ii}$ | 0.98 | 2.87 | 3.830 (4) | 167 |
| C51—H51*B*···Cl1$^{ii}$ | 0.98 | 2.85 | 3.809 (4) | 166 |
| C65—H65···Cl2$^{iii}$ | 0.95 | 2.91 | 3.514 (4) | 123 |

Symmetry codes: (i) $x+1/2, -y+1/2, -z+1$; (ii) $-x, y-1/2, -z+3/2$; (iii) $-x+1/2, -y+1, z+1/2$.