# metal-organic compounds

## Bis{1,2-bis[bis(3-methoxypropyl)phosphanyl]ethane-$\kappa^2 P,P'$}dichlorido-osmium(II)

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In the centrosymmetric title compound, $[OsCl_2(C_{18}H_{40}O_4P_2)_2]$, the $Os^{II}$ atom adopts a *trans*-$OsCl_2P_4$ geometry, arising from its coordination by two chelating diphosphane ligands and two chloride ions. One of the methoxy side chains of the ligand is disordered over two orientations in a 0.700 (6):0.300 (6) ratio.

## Related literature

For background to transition-metal dihydride complexes, see: Egbert *et al.* (2007); Heinekey *et al.* (2004); Miller *et al.* (2002); Szymczak & Tyler (2007); Szymczak *et al.* (2006).
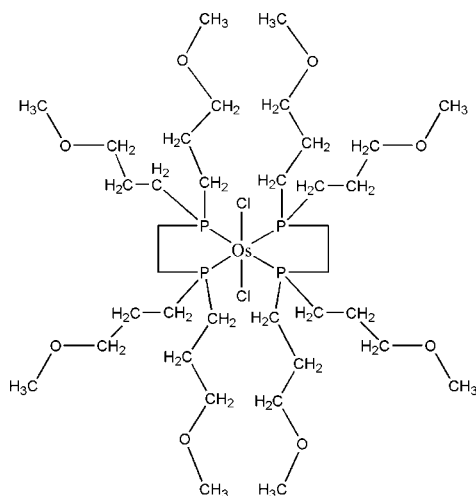


## Experimental

### Crystal data

| | |
|---|---|
| $[OsCl_2(C_{18}H_{40}O_4P_2)_2]$ | $V = 2335.0$ (9) Å$^3$ |
| $M_r = 1025.98$ | $Z = 2$ |
| Monoclinic, $P2_1/n$ | Mo $K\alpha$ radiation |
| $a = 12.667$ (3) Å | $\mu = 3.03$ mm$^{-1}$ |
| $b = 10.321$ (2) Å | $T = 173$ K |
| $c = 18.754$ (4) Å | $0.14 \times 0.10 \times 0.04$ mm |
| $\beta = 107.779$ (3)° | |

### Data collection

| | |
|---|---|
| Bruker APEX CCD diffractometer | 25950 measured reflections |
| Absorption correction: multi-scan (*SADABS*; Sheldrick, 1995) | 5333 independent reflections |
| $T_{min} = 0.677$, $T_{max} = 0.889$ | 4423 reflections with $I > 2\sigma(I)$ |
| | $R_{int} = 0.032$ |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)] = 0.024$ | 5 restraints |
| $wR(F^2) = 0.058$ | H-atom parameters constrained |
| $S = 1.04$ | $\Delta\rho_{max} = 0.87$ e Å$^{-3}$ |
| 5333 reflections | $\Delta\rho_{min} = -0.51$ e Å$^{-3}$ |
| 251 parameters | |

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

| | | | |
|---|---|---|---|
| Os1—P2 | 2.3383 (8) | Os1—Cl1 | 2.4515 (8) |
| Os1—P1 | 2.3434 (8) | | |

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

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# supporting information

## Bis{1,2-bis[bis(3-methoxypropyl)phosphanyl]ethane-$\kappa^2 P,P'$}dichloridoosmium(II)

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### S1. Comment

A consequence of the large d-orbitals in third-row transition metals is increased overlap to antibonding orbitals of ligands such as CO and $H_2$. Accordingly, when most third-row transition metal complexes are reacted with $H_2$, the product is often a dihydride complex (Egbert et al., 2007; Szymczak & Tyler, 2007). However, when the d-orbitals are not sufficiently electron rich to promote a complete oxidative-addition product, an arrested oxidative addition can result. The "arrested" intermediates belong to an unusual class of dihydrogen complexes with a bond distance between 1.1 Å and 1.5 Å (Heinekey et al., 2004). The H-H bond in these complexes sits in an unusually flat potential energy surface, and the H-H bond distance is consequently very sensitive to changes in the environment around the coordinated $\eta^2$-$H_2$ ligand. We hypothesized that small changes to the environment around the $H_2$ ligand could potentially be correlated with small energetic fluctuations, such as those that are typical of weak intermolecular forces such as hydrogen bonding.

Several osmium dihydrogen complexes have previously been shown to have H-H bond distances in the elongated regime (1.1 - 1.5 Å). For our study of hydrogen bonding of water to the $H_2$ ligand, we sought therefore to study hydrogen bonding to the $H_2$ ligand in the water-soluble trans-Os(DMeOPrPE)$_2$($H_2$)H$^+$ complex. (DMeOPrPE is the water-soluble, bidentate phosphine ligand 1,2-bis(bis(methoxypropyl)phosphanyl)ethane.) In prior work, we synthesized trans-Fe(DMeOPrPE)$_2$($H_2$)H$^+$ and trans-Ru(DMeOPrPE)$_2$($H_2$)H$^+$ by reaction of the trans-M(DMeOPrPE)$_2$Cl$_2$ complexes with $H_2$ (Miller et al., 2002, Szymczak et al., 2006). In order to synthesize the analogous $H_2$ complex of Os, we synthesized the trans-Os(DMeOPrPE)$_2$Cl$_2$ complex reported here.

The structure shows an octahedral coordination environment around osmium with trans-chloride ligands (Fig. 1). Crystallization of the osmium congener allowed a direct comparison of trans-M(DMeOPrPE)$_2$Cl$_2$ complexes down the group 8 triad (Table 1). The M-L bonds were found to increase substantially from iron to ruthenium with minimal elongation from ruthenium to osmium. This minimal change in going from ruthenium to osmium is consistent with a lanthanide contraction of the atomic radius.

### S2. Experimental

To a flask containing [OsCl$_6$][NEt$_4$]$_2$ (0.9114 g, 1.374 mmol) and NaBPh$_4$ (1.890 g, 5.523 mmol) was added a solution of DMeOPrPE (2.11 g, 5.524 mmol) in methanol (300 ml) followed by ethanol (365 ml). After heating to reflux for 16 h, the solution was deep purple with a white precipitate, and the solution was kept at reflux. After 4 days, the solution was clear yellow with a white solid on the flask walls. The solvent was removed and the residue was extracted with hot hexanes (3 x 30 ml) and diethyl ether (3 x 30 ml), leaving a yellow oil. Upon letting the oil stand for 24 h, yellow crystals of *trans*-[Os(DMeOPrPE)$_2$Cl$_2$] developed. Yield: 0.862 g (61%); {$^1$H} $^{31}$P NMR: d 8.6.

## S3. Refinement

The structure was solved using direct methods and refined with anisotropic thermal parameters for non-H atoms. H atoms were positioned geometrically and refined in a rigid group model, C—H = $1.2U_{eq}(C)$ and $1.5U_{eq}(C)$, respectively for –$CH_2$ and –$CH_3$ groups. In the molecule there are eight -$(CH_2)_3OCH_3$ terminal groups which are flexible and thermal parameters for atoms in these groups are significantly elongated. One of the –$CH_2OCH_3$ groups, C(17)O(4) C(18), is disordered over two positions in the ratio 0.700/0.300. Restrictions have been used in the refinement of this group; the typical values of the –$CH_2$—$CH_2$–, –$CH_2$—O– and –O—$CH_3$ bonds (1.524, 1.426 and 1.416 Å, respectively) have been used in the refinement as the targets for corresponding bond lengths.
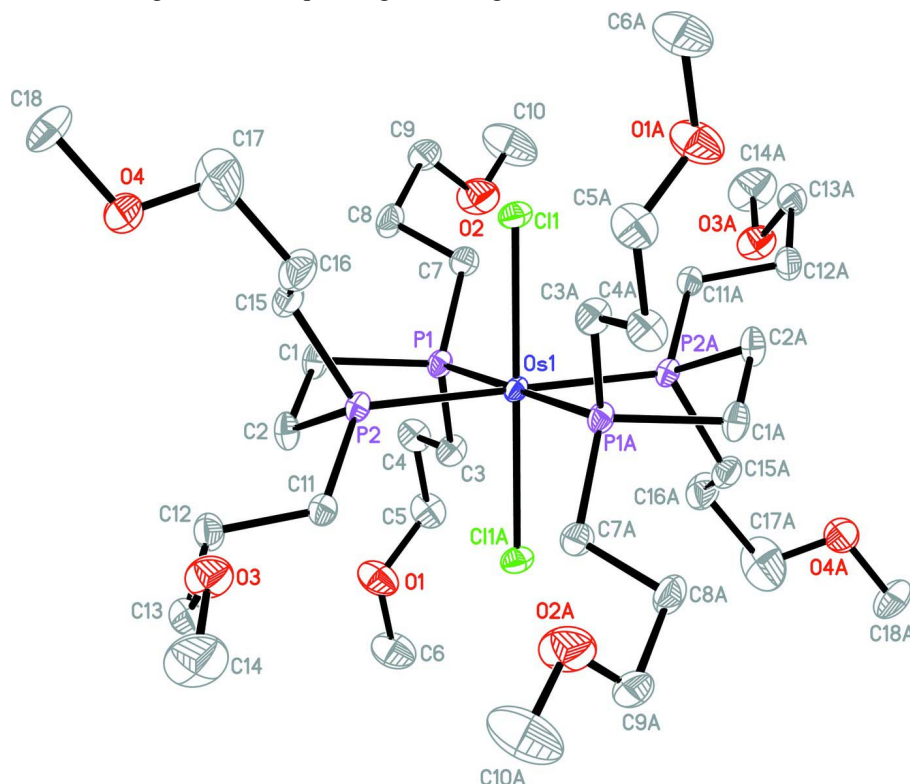


**Figure 1**

The structure of (I) with 50% probability displacement ellipsoids [Symmetry code (A): -x,-y,-z]. Only one position of the disordered C17/O4/C18 group is shown for clarity.

**Bis{1,2-bis[bis(3-methoxypropyl)phosphanyl]ethane-κ²P,P′}dichloridoosmium(II)**

*Crystal data*

| | |
|---|---|
| $[OsCl_2(C_{18}H_{40}O_4P_2)_2]$ | $F(000) = 1060$ |
| $M_r = 1025.98$ | $D_x = 1.459$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/n$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: -P 2yn | Cell parameters from 8415 reflections |
| $a = 12.667$ (3) Å | $\theta = 2.3–26.7°$ |
| $b = 10.321$ (2) Å | $\mu = 3.03$ mm$^{-1}$ |
| $c = 18.754$ (4) Å | $T = 173$ K |
| $\beta = 107.779$ (3)° | Plate, yellow |
| $V = 2335.0$ (9) Å$^3$ | 0.14 × 0.10 × 0.04 mm |
| $Z = 2$ | |

*Data collection*

| | |
|---|---|
| Bruker APEX CCD diffractometer | 25950 measured reflections |
| Radiation source: fine-focus sealed tube | 5333 independent reflections |
| Graphite monochromator | 4423 reflections with $I > 2\sigma(I)$ |
| phi and $\omega$ scans | $R_{int} = 0.032$ |
| Absorption correction: multi-scan | $\theta_{max} = 27.5°, \theta_{min} = 1.7°$ |
| ($SADABS$; Sheldrick, 1995) | $h = -16\rightarrow16$ |
| $T_{min} = 0.677, T_{max} = 0.889$ | $k = -13\rightarrow13$ |
| | $l = -23\rightarrow24$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | |
| $R[F^2 > 2\sigma(F^2)] = 0.024$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.058$ | |
| $S = 1.04$ | H-atom parameters constrained |
| 5333 reflections | $w = 1/[\sigma^2(F_o^2) + (0.028P)^2 + 0.9782P]$ |
| 251 parameters | where $P = (F_o^2 + 2F_c^2)/3$ |
| 5 restraints | $(\Delta/\sigma)_{max} < 0.001$ |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{max} = 0.87$ e Å$^{-3}$ |
| | $\Delta\rho_{min} = -0.51$ e Å$^{-3}$ |

*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.

**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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Os1 | 0.5000 | 0.5000 | 0.0000 | 0.02110 (5) | |
| Cl1 | 0.31396 (6) | 0.41077 (7) | −0.05728 (4) | 0.03191 (16) | |
| P1 | 0.57375 (6) | 0.32518 (7) | −0.04887 (4) | 0.02556 (15) | |
| P2 | 0.53170 (6) | 0.36384 (7) | 0.10408 (4) | 0.02567 (15) | |
| O1 | 0.9493 (2) | 0.2799 (3) | −0.02252 (14) | 0.0575 (7) | |
| O2 | 0.5043 (2) | 0.1314 (3) | −0.27322 (14) | 0.0666 (8) | |
| O3 | 0.5690 (2) | 0.4496 (3) | 0.35068 (13) | 0.0512 (6) | |
| C1 | 0.5965 (3) | 0.1882 (3) | 0.01729 (16) | 0.0354 (7) | |
| H1A | 0.5270 | 0.1384 | 0.0089 | 0.043* | |
| H1B | 0.6539 | 0.1295 | 0.0096 | 0.043* | |
| C2 | 0.6341 (3) | 0.2421 (3) | 0.09683 (16) | 0.0352 (7) | |
| H2A | 0.7082 | 0.2823 | 0.1076 | 0.042* | |
| H2B | 0.6386 | 0.1715 | 0.1334 | 0.042* | |
| C3 | 0.7111 (2) | 0.3487 (3) | −0.05918 (18) | 0.0355 (7) | |
| H3A | 0.7085 | 0.4293 | −0.0883 | 0.043* | |

| | | | | |
|---|---|---|---|---|
| H3B | 0.7632 | 0.3645 | −0.0086 | 0.043* |
| C4 | 0.7620 (3) | 0.2440 (3) | −0.09552 (18) | 0.0397 (7) |
| H4A | 0.7657 | 0.1617 | −0.0677 | 0.048* |
| H4B | 0.7141 | 0.2298 | −0.1475 | 0.048* |
| C5 | 0.8773 (3) | 0.2810 (4) | −0.09625 (19) | 0.0457 (8) |
| H5A | 0.9035 | 0.2188 | −0.1273 | 0.055* |
| H5B | 0.8759 | 0.3685 | −0.1182 | 0.055* |
| C6 | 1.0568 (3) | 0.3250 (5) | −0.0175 (2) | 0.0691 (12) |
| H6A | 1.1037 | 0.3219 | 0.0348 | 0.104* |
| H6B | 1.0521 | 0.4144 | −0.0358 | 0.104* |
| H6C | 1.0889 | 0.2699 | −0.0481 | 0.104* |
| C7 | 0.4913 (3) | 0.2555 (3) | −0.13885 (17) | 0.0336 (7) |
| H7A | 0.4127 | 0.2782 | −0.1465 | 0.040* |
| H7B | 0.5140 | 0.2990 | −0.1789 | 0.040* |
| C8 | 0.4975 (3) | 0.1093 (3) | −0.15003 (18) | 0.0410 (8) |
| H8A | 0.5761 | 0.0816 | −0.1339 | 0.049* |
| H8B | 0.4593 | 0.0638 | −0.1185 | 0.049* |
| C9 | 0.4444 (3) | 0.0725 (3) | −0.23097 (19) | 0.0455 (8) |
| H9A | 0.4455 | −0.0228 | −0.2367 | 0.055* |
| H9B | 0.3664 | 0.1021 | −0.2481 | 0.055* |
| C10 | 0.4635 (4) | 0.1030 (6) | −0.3488 (2) | 0.0944 (18) |
| H10A | 0.5089 | 0.1464 | −0.3755 | 0.142* |
| H10B | 0.3867 | 0.1333 | −0.3683 | 0.142* |
| H10C | 0.4659 | 0.0091 | −0.3560 | 0.142* |
| C11 | 0.5917 (2) | 0.4287 (3) | 0.19857 (16) | 0.0303 (6) |
| H11A | 0.6554 | 0.4842 | 0.1984 | 0.036* |
| H11B | 0.5356 | 0.4853 | 0.2097 | 0.036* |
| C12 | 0.6318 (3) | 0.3317 (3) | 0.26298 (17) | 0.0381 (7) |
| H12A | 0.5718 | 0.2690 | 0.2608 | 0.046* |
| H12B | 0.6959 | 0.2829 | 0.2571 | 0.046* |
| C13 | 0.6655 (3) | 0.3987 (3) | 0.33861 (18) | 0.0437 (8) |
| H13A | 0.7016 | 0.3361 | 0.3786 | 0.052* |
| H13B | 0.7187 | 0.4693 | 0.3393 | 0.052* |
| C14 | 0.5931 (5) | 0.5225 (4) | 0.4180 (3) | 0.0833 (16) |
| H14A | 0.5240 | 0.5559 | 0.4241 | 0.125* |
| H14B | 0.6419 | 0.5950 | 0.4157 | 0.125* |
| H14C | 0.6301 | 0.4667 | 0.4606 | 0.125* |
| C15 | 0.4218 (3) | 0.2540 (3) | 0.11326 (17) | 0.0374 (7) |
| H15A | 0.3810 | 0.2211 | 0.0628 | 0.045* |
| H15B | 0.4577 | 0.1787 | 0.1438 | 0.045* |
| C16 | 0.3385 (3) | 0.3093 (3) | 0.1475 (2) | 0.0509 (9) |
| H16A | 0.2935 | 0.3763 | 0.1140 | 0.061* |
| H16B | 0.3777 | 0.3508 | 0.1960 | 0.061* |
| C17 | 0.2609 (4) | 0.2002 (6) | 0.1602 (3) | 0.0890 (18) | 0.700 (6) |
| H17A | 0.1975 | 0.2396 | 0.1727 | 0.107* | 0.700 (6) |
| H17B | 0.2313 | 0.1495 | 0.1136 | 0.107* | 0.700 (6) |
| O4 | 0.3138 (3) | 0.1245 (3) | 0.2135 (2) | 0.0515 (12) | 0.700 (6) |
| C18 | 0.2400 (6) | 0.0305 (5) | 0.2282 (4) | 0.0579 (17) | 0.700 (6) |

| | | | | | |
|---|---|---|---|---|---|
| H18A | 0.2813 | −0.0264 | 0.2690 | 0.087* | 0.700 (6) |
| H18B | 0.2072 | −0.0213 | 0.1830 | 0.087* | 0.700 (6) |
| H18C | 0.1812 | 0.0747 | 0.2426 | 0.087* | 0.700 (6) |
| C17A | 0.2609 (4) | 0.2002 (6) | 0.1602 (3) | 0.0890 (18) | 0.300 (6) |
| H17C | 0.2140 | 0.1721 | 0.1101 | 0.107* | 0.300 (6) |
| H17D | 0.3090 | 0.1257 | 0.1825 | 0.107* | 0.300 (6) |
| O4A | 0.2061 (9) | 0.2156 (9) | 0.1940 (6) | 0.072 (4) | 0.300 (6) |
| C18A | 0.1540 (15) | 0.0978 (17) | 0.2088 (10) | 0.098 (7) | 0.300 (6) |
| H18D | 0.1048 | 0.1184 | 0.2387 | 0.146* | 0.300 (6) |
| H18E | 0.2112 | 0.0368 | 0.2364 | 0.146* | 0.300 (6) |
| H18F | 0.1106 | 0.0586 | 0.1612 | 0.146* | 0.300 (6) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Os1 | 0.02079 (8) | 0.01550 (8) | 0.02816 (8) | −0.00026 (6) | 0.00918 (6) | 0.00023 (6) |
| Cl1 | 0.0249 (3) | 0.0302 (4) | 0.0410 (4) | −0.0056 (3) | 0.0105 (3) | −0.0045 (3) |
| P1 | 0.0281 (4) | 0.0187 (3) | 0.0315 (4) | 0.0028 (3) | 0.0115 (3) | 0.0000 (3) |
| P2 | 0.0300 (4) | 0.0183 (3) | 0.0300 (4) | 0.0004 (3) | 0.0112 (3) | 0.0013 (3) |
| O1 | 0.0332 (13) | 0.091 (2) | 0.0499 (15) | 0.0038 (14) | 0.0149 (11) | 0.0043 (14) |
| O2 | 0.080 (2) | 0.078 (2) | 0.0400 (14) | −0.0198 (16) | 0.0150 (14) | −0.0137 (14) |
| O3 | 0.0617 (17) | 0.0534 (14) | 0.0407 (14) | −0.0105 (13) | 0.0189 (12) | −0.0129 (12) |
| C1 | 0.0489 (19) | 0.0217 (15) | 0.0398 (17) | 0.0087 (13) | 0.0196 (15) | 0.0028 (13) |
| C2 | 0.0464 (18) | 0.0263 (15) | 0.0345 (16) | 0.0116 (13) | 0.0146 (14) | 0.0071 (12) |
| C3 | 0.0338 (16) | 0.0309 (16) | 0.0464 (18) | 0.0029 (13) | 0.0188 (14) | −0.0018 (14) |
| C4 | 0.0377 (18) | 0.0457 (19) | 0.0392 (18) | 0.0094 (15) | 0.0169 (15) | −0.0033 (15) |
| C5 | 0.0375 (18) | 0.060 (2) | 0.047 (2) | 0.0101 (16) | 0.0230 (16) | −0.0002 (17) |
| C6 | 0.036 (2) | 0.097 (4) | 0.074 (3) | 0.004 (2) | 0.016 (2) | −0.002 (3) |
| C7 | 0.0354 (17) | 0.0254 (15) | 0.0382 (17) | 0.0027 (12) | 0.0088 (14) | −0.0059 (13) |
| C8 | 0.057 (2) | 0.0219 (16) | 0.0427 (18) | −0.0017 (14) | 0.0134 (16) | −0.0029 (13) |
| C9 | 0.051 (2) | 0.0304 (18) | 0.050 (2) | −0.0037 (15) | 0.0078 (17) | −0.0116 (15) |
| C10 | 0.074 (3) | 0.160 (6) | 0.044 (3) | 0.007 (3) | 0.011 (2) | −0.018 (3) |
| C11 | 0.0334 (16) | 0.0243 (16) | 0.0323 (15) | 0.0008 (12) | 0.0086 (13) | 0.0014 (12) |
| C12 | 0.0465 (19) | 0.0299 (17) | 0.0347 (16) | 0.0052 (14) | 0.0074 (14) | 0.0044 (13) |
| C13 | 0.051 (2) | 0.0391 (19) | 0.0338 (17) | −0.0034 (16) | 0.0019 (15) | 0.0051 (14) |
| C14 | 0.115 (5) | 0.076 (3) | 0.061 (3) | −0.019 (3) | 0.031 (3) | −0.033 (2) |
| C15 | 0.0468 (19) | 0.0304 (17) | 0.0363 (17) | −0.0084 (14) | 0.0148 (15) | 0.0023 (13) |
| C16 | 0.046 (2) | 0.048 (2) | 0.065 (2) | −0.0052 (17) | 0.0256 (18) | 0.0135 (18) |
| C17 | 0.080 (3) | 0.128 (5) | 0.076 (3) | 0.005 (3) | 0.048 (3) | 0.044 (3) |
| O4 | 0.041 (2) | 0.058 (2) | 0.056 (2) | −0.0074 (17) | 0.0166 (17) | 0.0144 (18) |
| C18 | 0.059 (4) | 0.047 (3) | 0.080 (4) | −0.018 (3) | 0.042 (4) | 0.005 (3) |
| C17A | 0.080 (3) | 0.128 (5) | 0.076 (3) | 0.005 (3) | 0.048 (3) | 0.044 (3) |
| O4A | 0.077 (7) | 0.062 (6) | 0.077 (7) | −0.007 (5) | 0.024 (6) | 0.012 (5) |
| C18A | 0.076 (12) | 0.099 (14) | 0.105 (13) | −0.061 (11) | 0.009 (10) | 0.042 (11) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Os1—P2 | 2.3383 (8) | C8—C9 | 1.509 (4) |
| Os1—P2ⁱ | 2.3383 (8) | C8—H8A | 0.9900 |
| Os1—P1ⁱ | 2.3434 (8) | C8—H8B | 0.9900 |
| Os1—P1 | 2.3434 (8) | C9—H9A | 0.9900 |
| Os1—Cl1 | 2.4515 (8) | C9—H9B | 0.9900 |
| Os1—Cl1ⁱ | 2.4515 (8) | C10—H10A | 0.9800 |
| P1—C3 | 1.825 (3) | C10—H10B | 0.9800 |
| P1—C7 | 1.838 (3) | C10—H10C | 0.9800 |
| P1—C1 | 1.845 (3) | C11—C12 | 1.531 (4) |
| P2—C11 | 1.829 (3) | C11—H11A | 0.9900 |
| P2—C2 | 1.840 (3) | C11—H11B | 0.9900 |
| P2—C15 | 1.844 (3) | C12—C13 | 1.517 (4) |
| O1—C5 | 1.405 (4) | C12—H12A | 0.9900 |
| O1—C6 | 1.414 (4) | C12—H12B | 0.9900 |
| O2—C10 | 1.384 (5) | C13—H13A | 0.9900 |
| O2—C9 | 1.392 (4) | C13—H13B | 0.9900 |
| O3—C13 | 1.412 (4) | C14—H14A | 0.9800 |
| O3—C14 | 1.420 (5) | C14—H14B | 0.9800 |
| C1—C2 | 1.525 (4) | C14—H14C | 0.9800 |
| C1—H1A | 0.9900 | C15—C16 | 1.506 (5) |
| C1—H1B | 0.9900 | C15—H15A | 0.9900 |
| C2—H2A | 0.9900 | C15—H15B | 0.9900 |
| C2—H2B | 0.9900 | C16—C17 | 1.560 (6) |
| C3—C4 | 1.521 (4) | C16—H16A | 0.9900 |
| C3—H3A | 0.9900 | C16—H16B | 0.9900 |
| C3—H3B | 0.9900 | C17—O4 | 1.283 (5) |
| C4—C5 | 1.514 (4) | C17—H17A | 0.9900 |
| C4—H4A | 0.9900 | C17—H17B | 0.9900 |
| C4—H4B | 0.9900 | O4—C18 | 1.431 (6) |
| C5—H5A | 0.9900 | C18—H18A | 0.9800 |
| C5—H5B | 0.9900 | C18—H18B | 0.9800 |
| C6—H6A | 0.9800 | C18—H18C | 0.9800 |
| C6—H6B | 0.9800 | O4A—C18A | 1.451 (13) |
| C6—H6C | 0.9800 | C18A—H18D | 0.9800 |
| C7—C8 | 1.528 (4) | C18A—H18E | 0.9800 |
| C7—H7A | 0.9900 | C18A—H18F | 0.9800 |
| C7—H7B | 0.9900 | | |
| | | | |
| P2—Os1—P2ⁱ | 180.00 (4) | C9—C8—H8A | 109.4 |
| P2—Os1—P1ⁱ | 97.10 (3) | C7—C8—H8A | 109.4 |
| P2ⁱ—Os1—P1ⁱ | 82.90 (3) | C9—C8—H8B | 109.4 |
| P2—Os1—P1 | 82.90 (3) | C7—C8—H8B | 109.4 |
| P2ⁱ—Os1—P1 | 97.10 (3) | H8A—C8—H8B | 108.0 |
| P1ⁱ—Os1—P1 | 180.00 (3) | O2—C9—C8 | 108.1 (3) |
| P2—Os1—Cl1 | 92.13 (3) | O2—C9—H9A | 110.1 |
| P2ⁱ—Os1—Cl1 | 87.87 (3) | C8—C9—H9A | 110.1 |

| | | | |
|---|---|---|---|
| P1ⁱ—Os1—Cl1 | 90.91 (3) | O2—C9—H9B | 110.1 |
| P1—Os1—Cl1 | 89.09 (3) | C8—C9—H9B | 110.1 |
| P2—Os1—Cl1ⁱ | 87.87 (3) | H9A—C9—H9B | 108.4 |
| P2ⁱ—Os1—Cl1ⁱ | 92.13 (3) | O2—C10—H10A | 109.5 |
| P1ⁱ—Os1—Cl1ⁱ | 89.09 (3) | O2—C10—H10B | 109.5 |
| P1—Os1—Cl1ⁱ | 90.91 (3) | H10A—C10—H10B | 109.5 |
| Cl1—Os1—Cl1ⁱ | 180.0 | O2—C10—H10C | 109.5 |
| C3—P1—C7 | 104.32 (15) | H10A—C10—H10C | 109.5 |
| C3—P1—C1 | 102.40 (15) | H10B—C10—H10C | 109.5 |
| C7—P1—C1 | 104.24 (14) | C12—C11—P2 | 117.7 (2) |
| C3—P1—Os1 | 116.50 (10) | C12—C11—H11A | 107.9 |
| C7—P1—Os1 | 118.55 (10) | P2—C11—H11A | 107.9 |
| C1—P1—Os1 | 109.03 (10) | C12—C11—H11B | 107.9 |
| C11—P2—C2 | 103.16 (14) | P2—C11—H11B | 107.9 |
| C11—P2—C15 | 103.69 (14) | H11A—C11—H11B | 107.2 |
| C2—P2—C15 | 98.98 (15) | C13—C12—C11 | 111.8 (2) |
| C11—P2—Os1 | 120.25 (10) | C13—C12—H12A | 109.3 |
| C2—P2—Os1 | 107.11 (10) | C11—C12—H12A | 109.3 |
| C15—P2—Os1 | 120.36 (11) | C13—C12—H12B | 109.3 |
| C5—O1—C6 | 112.7 (3) | C11—C12—H12B | 109.3 |
| C10—O2—C9 | 113.1 (3) | H12A—C12—H12B | 107.9 |
| C13—O3—C14 | 112.2 (3) | O3—C13—C12 | 108.0 (3) |
| C2—C1—P1 | 108.5 (2) | O3—C13—H13A | 110.1 |
| C2—C1—H1A | 110.0 | C12—C13—H13A | 110.1 |
| P1—C1—H1A | 110.0 | O3—C13—H13B | 110.1 |
| C2—C1—H1B | 110.0 | C12—C13—H13B | 110.1 |
| P1—C1—H1B | 110.0 | H13A—C13—H13B | 108.4 |
| H1A—C1—H1B | 108.4 | O3—C14—H14A | 109.5 |
| C1—C2—P2 | 107.7 (2) | O3—C14—H14B | 109.5 |
| C1—C2—H2A | 110.2 | H14A—C14—H14B | 109.5 |
| P2—C2—H2A | 110.2 | O3—C14—H14C | 109.5 |
| C1—C2—H2B | 110.2 | H14A—C14—H14C | 109.5 |
| P2—C2—H2B | 110.2 | H14B—C14—H14C | 109.5 |
| H2A—C2—H2B | 108.5 | C16—C15—P2 | 117.0 (2) |
| C4—C3—P1 | 120.1 (2) | C16—C15—H15A | 108.0 |
| C4—C3—H3A | 107.3 | P2—C15—H15A | 108.0 |
| P1—C3—H3A | 107.3 | C16—C15—H15B | 108.0 |
| C4—C3—H3B | 107.3 | P2—C15—H15B | 108.0 |
| P1—C3—H3B | 107.3 | H15A—C15—H15B | 107.3 |
| H3A—C3—H3B | 106.9 | C15—C16—C17 | 110.3 (3) |
| C5—C4—C3 | 111.5 (3) | C15—C16—H16A | 109.6 |
| C5—C4—H4A | 109.3 | C17—C16—H16A | 109.6 |
| C3—C4—H4A | 109.3 | C15—C16—H16B | 109.6 |
| C5—C4—H4B | 109.3 | C17—C16—H16B | 109.6 |
| C3—C4—H4B | 109.3 | H16A—C16—H16B | 108.1 |
| H4A—C4—H4B | 108.0 | O4—C17—C16 | 110.8 (4) |
| O1—C5—C4 | 109.1 (3) | O4—C17—H17A | 109.5 |
| O1—C5—H5A | 109.9 | C16—C17—H17A | 109.5 |

| | | | |
|---|---|---|---|
| C4—C5—H5A | 109.9 | O4—C17—H17B | 109.5 |
| O1—C5—H5B | 109.9 | C16—C17—H17B | 109.5 |
| C4—C5—H5B | 109.9 | H17A—C17—H17B | 108.1 |
| H5A—C5—H5B | 108.3 | C17—O4—C18 | 110.3 (4) |
| O1—C6—H6A | 109.5 | O4—C18—H18A | 109.5 |
| O1—C6—H6B | 109.5 | O4—C18—H18B | 109.5 |
| H6A—C6—H6B | 109.5 | H18A—C18—H18B | 109.5 |
| O1—C6—H6C | 109.5 | O4—C18—H18C | 109.5 |
| H6A—C6—H6C | 109.5 | H18A—C18—H18C | 109.5 |
| H6B—C6—H6C | 109.5 | H18B—C18—H18C | 109.5 |
| C8—C7—P1 | 118.3 (2) | O4A—C18A—H18D | 109.5 |
| C8—C7—H7A | 107.7 | O4A—C18A—H18E | 109.5 |
| P1—C7—H7A | 107.7 | H18D—C18A—H18E | 109.5 |
| C8—C7—H7B | 107.7 | O4A—C18A—H18F | 109.5 |
| P1—C7—H7B | 107.7 | H18D—C18A—H18F | 109.5 |
| H7A—C7—H7B | 107.1 | H18E—C18A—H18F | 109.5 |
| C9—C8—C7 | 111.2 (3) | | |
| | | | |
| P2—Os1—P1—C3 | −109.55 (12) | C7—P1—C1—C2 | −163.7 (2) |
| P2[i]—Os1—P1—C3 | 70.45 (12) | Os1—P1—C1—C2 | −36.2 (2) |
| P1[i]—Os1—P1—C3 | 5 (47) | P1—C1—C2—P2 | 53.3 (2) |
| Cl1—Os1—P1—C3 | 158.19 (12) | C11—P2—C2—C1 | −175.8 (2) |
| Cl1[i]—Os1—P1—C3 | −21.81 (12) | C15—P2—C2—C1 | 77.8 (2) |
| P2—Os1—P1—C7 | 124.59 (12) | Os1—P2—C2—C1 | −47.9 (2) |
| P2[i]—Os1—P1—C7 | −55.41 (12) | C7—P1—C3—C4 | −43.0 (3) |
| P1[i]—Os1—P1—C7 | −121 (47) | C1—P1—C3—C4 | 65.5 (3) |
| Cl1—Os1—P1—C7 | 32.34 (12) | Os1—P1—C3—C4 | −175.7 (2) |
| Cl1[i]—Os1—P1—C7 | −147.66 (12) | P1—C3—C4—C5 | −178.2 (2) |
| P2—Os1—P1—C1 | 5.65 (11) | C6—O1—C5—C4 | −174.6 (3) |
| P2[i]—Os1—P1—C1 | −174.35 (11) | C3—C4—C5—O1 | 68.8 (4) |
| P1[i]—Os1—P1—C1 | 120 (47) | C3—P1—C7—C8 | 83.9 (3) |
| Cl1—Os1—P1—C1 | −86.61 (11) | C1—P1—C7—C8 | −23.2 (3) |
| Cl1[i]—Os1—P1—C1 | 93.39 (11) | Os1—P1—C7—C8 | −144.6 (2) |
| P2[i]—Os1—P2—C11 | 88 (100) | P1—C7—C8—C9 | −168.4 (2) |
| P1[i]—Os1—P2—C11 | −43.13 (12) | C10—O2—C9—C8 | 179.3 (4) |
| P1—Os1—P2—C11 | 136.87 (12) | C7—C8—C9—O2 | 62.8 (4) |
| Cl1—Os1—P2—C11 | −134.31 (12) | C2—P2—C11—C12 | −49.0 (3) |
| Cl1[i]—Os1—P2—C11 | 45.69 (12) | C15—P2—C11—C12 | 53.8 (3) |
| P2[i]—Os1—P2—C2 | −29 (100) | Os1—P2—C11—C12 | −168.09 (19) |
| P1[i]—Os1—P2—C2 | −160.21 (11) | P2—C11—C12—C13 | −172.6 (2) |
| P1—Os1—P2—C2 | 19.79 (11) | C14—O3—C13—C12 | −175.8 (3) |
| Cl1—Os1—P2—C2 | 108.61 (11) | C11—C12—C13—O3 | 68.3 (3) |
| Cl1[i]—Os1—P2—C2 | −71.39 (11) | C11—P2—C15—C16 | 52.9 (3) |
| P2[i]—Os1—P2—C15 | −141 (100) | C2—P2—C15—C16 | 158.9 (3) |
| P1[i]—Os1—P2—C15 | 88.10 (12) | Os1—P2—C15—C16 | −85.1 (3) |
| P1—Os1—P2—C15 | −91.90 (12) | P2—C15—C16—C17 | −173.0 (3) |
| Cl1—Os1—P2—C15 | −3.08 (12) | C15—C16—C17—O4 | 70.8 (5) |

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|---|---|---|---|
| Cl1$^i$—Os1—P2—C15 | 176.92 (12) | C16—C17—O4—C18 | 175.9 (4) |
| C3—P1—C1—C2 | 87.8 (2) | | |

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