

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Dichlorido(η^4 -cycloocta-1,5-diene)bis-(triphenylphosphine)osmium(II)

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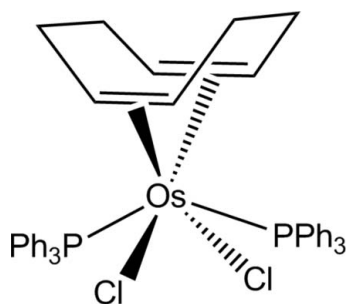
Received 14 September 2009; accepted 18 September 2009

Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.008$ Å; R factor = 0.022; wR factor = 0.044; data-to-parameter ratio = 12.5.

The Os^{II} atom in the title compound, $[\text{OsCl}_2(\text{C}_8\text{H}_{12})(\text{C}_{18}\text{H}_{15}\text{P})_2]$, is located on a crystallographic twofold axis and adopts a distorted octahedral coordination geometry. The two triphenylphosphine ligands are *trans* to each other, while the two chlorine ligands are *cis*-disposed. The coordination is completed by the cyclooctadiene (COD) ligand with bonding to the two olefin double bonds. The C=C bond has a length of 1.403 (6) Å, which is significantly longer than a free olefinic double bond (≈ 1.34 Å).

Related literature

For general background to Ru^{II} and Os^{II} COD complexes, see: Bennett & Wilkinson (1959); Albers *et al.* (1989); Cucullu *et al.* (1999); Coalter & Caulton (2001); Alvarez *et al.* (2001); Winkhaus *et al.* (1966); Schrock *et al.* (1974); Dickinson & Girolami (2006). For C=C bond lengths for free olefinic double bonds, see: Orpen *et al.* (1989). For related COD-coordinated Os^{II} complexes, see: Esteruelas *et al.* (2006); Dickinson & Girolami (2006).



Experimental

Crystal data

 $[\text{OsCl}_2(\text{C}_8\text{H}_{12})(\text{C}_{18}\text{H}_{15}\text{P})_2]$
 $M_r = 893.82$ Orthorhombic, $Fdd2$ $a = 39.6505$ (15) Å $b = 10.4393$ (5) Å $c = 17.6248$ (8) Å $V = 7295.3$ (6) Å³ $Z = 8$ Mo $K\alpha$ radiation $\mu = 3.76$ mm⁻¹ $T = 173$ K $0.15 \times 0.15 \times 0.12$ mm

Data collection

Oxford Diffraction Gemini S Ultra diffractometer

Absorption correction: multi-scan

(CrysAlis RED; Oxford

Diffraction, 2008)

 $T_{\min} = 0.860$, $T_{\max} = 1.000$

6965 measured reflections

2778 independent reflections

2546 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.033$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.022$ $wR(F^2) = 0.044$ $S = 1.00$

2778 reflections

222 parameters

1 restraint

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 1.39$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.74$ e Å⁻³

Absolute structure: Flack (1983),

937 Friedel pairs

Flack parameter: 0.009 (6)

Table 1

Selected bond lengths (Å).

| | | | |
|---------|-----------|---------|-------------|
| Os1—C2 | 2.169 (5) | Os1—Cl1 | 2.4429 (12) |
| Os1—Cl1 | 2.195 (5) | Os1—P1 | 2.5031 (12) |

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2008); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2008); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors acknowledge financial support from the Young Talent Project of Fujian Provincial Department of Science & Technology (grant No. 2007 F3095).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2258).

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Acta Cryst. (2009). E65, m1242 [doi:10.1107/S1600536809037817]

Dichlorido(η^4 -cycloocta-1,5-diene)bis(triphenylphosphine)osmium(II)

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Comment

The ruthenium polymer $[\text{RuCl}_2(\text{COD})]_x$ (COD = cycloocta-1,5-diene), which can be readily prepared in 30–40% yield from $\text{RuCl}_3 \cdot 3\text{H}_2\text{O}$ and COD in boiling ethanol (Bennett & Wilkinson, 1959; Albers *et al.*, 1989), has proved to be a useful precursor for a wide variety of ruthenium compounds (Cucullu *et al.*, 1999; Coalter & Caulton, 2001; Alvarez *et al.*, 2001). Although the analogous osmium polymer $[\text{OsCl}_2(\text{COD})]_x$ is also known, its utility as a starting material has remained relatively unexplored partially due to the difficulty in its preparation (Winkhaus *et al.*, 1966; Schrock *et al.*, 1974; Dickinson & Girolami, 2006). In our search for other potential precursors for the synthesis of osmium compounds, we have prepared $[\text{OsCl}_2(\eta^4\text{-COD})(\text{PPh}_3)_2]$ readily from the reaction of $\text{OsCl}_2(\text{PPh}_3)_3$ with COD.

The title compound crystallizes in the non-centrosymmetric orthorhombic space group *Fdd2*. As shown in Fig.1, the structure possesses a crystallographic 2-fold axis passing through the osmium atom, thus the asymmetric unit contains half of a molecule. The Os^{II} centre adopts a distorted octahedral geometry with the two triphenylphosphine ligands *trans* to each other ($\text{P}(1)\text{—Os}(1)\text{—P}(1\text{ A})$ 148.31 (6)°), while the two chlorine ligands are *cis*-disposed ($\text{Cl}(1)\text{—Os}(1)\text{—Cl}(1\text{ A})$ 103.69 (6)°) (symmetry code: $-x + 1/2, -y + 1/2, z$). The coordination is completed by the two olefin double bonds of the cyclooctadiene ligand. The $\text{Os}(1)\text{—C}(1)$ and $\text{Os}(1)\text{—C}(2)$ bond lengths (2.195 (5) and 2.168 (5) Å, respectively) are similar to the related Os—C bond lengths reported for other COD coordinated Os^{II} complexes such as $[\text{H}(\text{EtOH})_2\{\text{OsCl}(\eta^4\text{-COD})\}_2(\mu\text{-H})(\mu\text{-Cl})_2]$ (2.129–2.152 Å) (Esteruelas *et al.*, 2006) and $\text{TpOs}(\eta^4\text{-COD})\text{OMe}$ (Tp = trispyrazolylborate) (2.141–2.198 Å) (Dickinson & Girolami, 2006). The $\text{C}(1)\text{—C}(2)$ (1.403 (6) Å) bond length is significantly longer than a free olefinic double bond (≈ 1.34 Å) (Orpen *et al.*, 1989) and is typical for a coordinated C=C double bond, which is also close to the C=C bond lengths found in $[\text{H}(\text{EtOH})_2\{\text{OsCl}(\eta^4\text{-COD})\}_2(\mu\text{-H})(\mu\text{-Cl})_2]$ (1.393–1.422 Å) and $\text{TpOs}(\eta^4\text{-COD})\text{OMe}$ (1.396 (5) and 1.399 (5) Å).

Experimental

To a solution of $\text{OsCl}_2(\text{PPh}_3)_3$ (0.52 g, 0.50 mmol) in toluene (10 ml) under nitrogen atmosphere was added cycloocta-1,5-diene (0.20 ml, 2.5 mmol). The reaction mixture was stirred at room temperature for 30 h to give the title compound as large amount of a yellow precipitate. The solid was collected by filtration, washed with toluene (2×5 ml) and diethyl ether (3×5 ml), and dried under vacuum. Yield: 0.38 g, 85%. Crystals suitable for X-ray analysis were obtained by layering a solution of the title compound with a solution of chloroform and hexane.

Refinement

All non-hydrogen atoms were refined anisotropically. The hydrogen atoms were positioned geometrically ($\text{C—H} = 0.95, 1.00$ or 0.99 Å for phenyl, tertiary or methylene H atoms, respectively) and were included in the refinement in the riding

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model approximation. The displacement parameters of H atoms were set to $1.2U_{\text{eq}}(\text{C})$. In the final Fourier map the highest peak is 0.99 Å from atom Os1 and the deepest hole is 1.86 Å from atom C15.

Figures

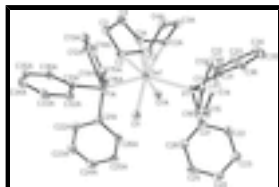


Fig. 1. The structure of $[\text{OsCl}_2(\text{C}_8\text{H}_{12})(\text{C}_{18}\text{H}_{15}\text{P})_2]$ showing 40% probability displacement ellipsoids. H atoms have been omitted for clarity. Atoms with symmetry code letter A are related by $-x + 1/2, -y + 1/2, z$.

Dichlorido(η^4 -cycloocta-1,5-diene)bis(triphenylphosphine)osmium(II)

Crystal data

$[\text{OsCl}_2(\text{C}_8\text{H}_{12})(\text{PC}_{18}\text{H}_{15})_2]$

$M_r = 893.82$

Orthorhombic, $Fdd2$

Hall symbol: $F\ 2\ -2d$

$a = 39.6505\ (15)\ \text{\AA}$

$b = 10.4393\ (5)\ \text{\AA}$

$c = 17.6248\ (8)\ \text{\AA}$

$V = 7295.3\ (6)\ \text{\AA}^3$

$Z = 8$

$F_{000} = 3568$

$D_x = 1.628\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 5605 reflections

$\theta = 2.3\text{--}32.5^\circ$

$\mu = 3.76\ \text{mm}^{-1}$

$T = 173\ \text{K}$

Block, light yellow

$0.15 \times 0.15 \times 0.12\ \text{mm}$

Data collection

Oxford Diffraction Gemini S Ultra diffractometer

Radiation source: Enhance (Mo) X-ray Source

Monochromator: graphite

Detector resolution: $16.1930\ \text{pixels mm}^{-1}$

$T = 173\ \text{K}$

ω scans

Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2008)

$T_{\text{min}} = 0.860, T_{\text{max}} = 1.000$

6965 measured reflections

2778 independent reflections

2546 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.033$

$\theta_{\text{max}} = 26.0^\circ$

$\theta_{\text{min}} = 2.3^\circ$

$h = -48 \rightarrow 48$

$k = -12 \rightarrow 12$

$l = -21 \rightarrow 16$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.022$

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0184P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

| | |
|--|--|
| $wR(F^2) = 0.044$ | $(\Delta/\sigma)_{\max} = 0.001$ |
| $S = 1.00$ | $\Delta\rho_{\max} = 1.39 \text{ e } \text{\AA}^{-3}$ |
| 2778 reflections | $\Delta\rho_{\min} = -0.74 \text{ e } \text{\AA}^{-3}$ |
| 222 parameters | Extinction correction: none |
| 1 restraint | Absolute structure: Flack (1983), 937 Friedel pairs |
| Primary atom site location: structure-invariant direct methods | Flack parameter: 0.009 (6) |
| Secondary atom site location: difference Fourier map | |

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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|--------------|---------------|----------------------------------|
| Os1 | 0.2500 | 0.2500 | 0.134570 (16) | 0.01584 (6) |
| C11 | 0.27183 (3) | 0.08574 (11) | 0.22020 (7) | 0.0225 (3) |
| P1 | 0.19622 (3) | 0.14284 (12) | 0.17335 (8) | 0.0183 (3) |
| C1 | 0.25093 (11) | 0.1050 (4) | 0.0444 (3) | 0.0232 (10) |
| H1A | 0.2499 | 0.0145 | 0.0629 | 0.028* |
| C2 | 0.28281 (11) | 0.1625 (5) | 0.0509 (3) | 0.0203 (10) |
| H2A | 0.3005 | 0.1053 | 0.0728 | 0.024* |
| C3 | 0.29572 (11) | 0.2541 (5) | -0.0092 (3) | 0.0243 (10) |
| H3A | 0.2991 | 0.2067 | -0.0572 | 0.029* |
| H3B | 0.3179 | 0.2882 | 0.0069 | 0.029* |
| C4 | 0.27153 (12) | 0.3668 (5) | -0.0234 (3) | 0.0279 (12) |
| H4A | 0.2849 | 0.4442 | -0.0357 | 0.033* |
| H4B | 0.2572 | 0.3468 | -0.0678 | 0.033* |
| C11 | 0.19375 (10) | -0.0268 (4) | 0.1464 (3) | 0.0222 (11) |
| C12 | 0.18371 (11) | -0.0649 (5) | 0.0742 (3) | 0.0263 (12) |
| H12A | 0.1759 | -0.0021 | 0.0393 | 0.032* |
| C13 | 0.18481 (12) | -0.1911 (5) | 0.0520 (4) | 0.0342 (13) |
| H13A | 0.1779 | -0.2156 | 0.0024 | 0.041* |
| C14 | 0.19615 (15) | -0.2814 (6) | 0.1031 (5) | 0.0346 (18) |
| H14A | 0.1969 | -0.3689 | 0.0885 | 0.041* |
| C15 | 0.20651 (14) | -0.2471 (6) | 0.1754 (5) | 0.0349 (18) |
| H15A | 0.2142 | -0.3107 | 0.2099 | 0.042* |
| C16 | 0.20554 (11) | -0.1196 (4) | 0.1973 (3) | 0.0257 (12) |
| H16A | 0.2129 | -0.0952 | 0.2465 | 0.031* |

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| | | | | |
|------|--------------|------------|------------|-------------|
| C21 | 0.18731 (10) | 0.1237 (4) | 0.2762 (3) | 0.0204 (10) |
| C22 | 0.15626 (12) | 0.0737 (5) | 0.2961 (3) | 0.0270 (12) |
| H22A | 0.1395 | 0.0618 | 0.2582 | 0.032* |
| C23 | 0.14915 (12) | 0.0407 (5) | 0.3707 (4) | 0.0318 (14) |
| H23A | 0.1276 | 0.0077 | 0.3835 | 0.038* |
| C24 | 0.17309 (13) | 0.0556 (5) | 0.4255 (3) | 0.0317 (13) |
| H24A | 0.1685 | 0.0310 | 0.4764 | 0.038* |
| C25 | 0.20399 (12) | 0.1068 (5) | 0.4063 (3) | 0.0306 (13) |
| H25A | 0.2207 | 0.1182 | 0.4444 | 0.037* |
| C26 | 0.21088 (12) | 0.1414 (5) | 0.3332 (3) | 0.0259 (12) |
| H26A | 0.2322 | 0.1780 | 0.3213 | 0.031* |
| C31 | 0.15530 (10) | 0.2115 (4) | 0.1440 (3) | 0.0191 (12) |
| C32 | 0.14963 (12) | 0.3414 (5) | 0.1543 (3) | 0.0240 (12) |
| H32A | 0.1670 | 0.3930 | 0.1754 | 0.029* |
| C33 | 0.11927 (12) | 0.3978 (5) | 0.1344 (4) | 0.0305 (13) |
| H33A | 0.1161 | 0.4873 | 0.1414 | 0.037* |
| C34 | 0.09364 (12) | 0.3237 (6) | 0.1046 (3) | 0.0323 (14) |
| H34A | 0.0731 | 0.3622 | 0.0889 | 0.039* |
| C35 | 0.09823 (12) | 0.1943 (6) | 0.0978 (3) | 0.0304 (13) |
| H35A | 0.0802 | 0.1423 | 0.0800 | 0.037* |
| C36 | 0.12872 (12) | 0.1382 (5) | 0.1165 (3) | 0.0246 (14) |
| H36A | 0.1315 | 0.0484 | 0.1106 | 0.029* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|--------------|--------------|--------------|------------|-------------|
| Os1 | 0.01417 (9) | 0.01427 (10) | 0.01910 (11) | 0.00191 (17) | 0.000 | 0.000 |
| Cl1 | 0.0193 (5) | 0.0201 (6) | 0.0280 (7) | 0.0041 (4) | 0.0005 (5) | 0.0053 (6) |
| P1 | 0.0167 (5) | 0.0157 (6) | 0.0224 (7) | 0.0016 (5) | 0.0000 (5) | -0.0006 (6) |
| C1 | 0.027 (2) | 0.016 (2) | 0.027 (3) | 0.004 (2) | 0.007 (3) | -0.005 (2) |
| C2 | 0.022 (2) | 0.019 (3) | 0.019 (3) | -0.0009 (19) | 0.000 (2) | -0.005 (2) |
| C3 | 0.023 (2) | 0.027 (3) | 0.023 (2) | 0.001 (2) | 0.000 (2) | -0.003 (3) |
| C4 | 0.030 (2) | 0.026 (3) | 0.028 (3) | -0.002 (2) | -0.003 (2) | 0.003 (3) |
| C11 | 0.0164 (19) | 0.016 (2) | 0.034 (3) | -0.0028 (16) | 0.004 (2) | -0.007 (2) |
| C12 | 0.024 (2) | 0.023 (3) | 0.031 (3) | -0.001 (2) | 0.001 (2) | -0.003 (2) |
| C13 | 0.030 (3) | 0.031 (3) | 0.041 (3) | 0.004 (2) | 0.002 (3) | -0.014 (3) |
| C14 | 0.027 (3) | 0.017 (3) | 0.060 (5) | 0.004 (2) | 0.005 (3) | -0.011 (3) |
| C15 | 0.022 (3) | 0.027 (3) | 0.055 (5) | 0.008 (2) | 0.000 (3) | 0.007 (4) |
| C16 | 0.021 (2) | 0.020 (3) | 0.035 (3) | -0.0002 (19) | -0.003 (2) | -0.001 (2) |
| C21 | 0.021 (2) | 0.019 (2) | 0.022 (3) | 0.0058 (18) | 0.005 (2) | 0.005 (2) |
| C22 | 0.023 (2) | 0.028 (3) | 0.030 (3) | 0.000 (2) | -0.001 (2) | 0.005 (3) |
| C23 | 0.029 (2) | 0.031 (3) | 0.035 (4) | -0.0005 (18) | 0.005 (3) | 0.007 (3) |
| C24 | 0.043 (3) | 0.029 (3) | 0.024 (3) | 0.008 (2) | 0.004 (3) | 0.005 (3) |
| C25 | 0.030 (3) | 0.039 (3) | 0.023 (3) | 0.004 (2) | -0.002 (2) | -0.003 (3) |
| C26 | 0.025 (2) | 0.025 (3) | 0.028 (3) | 0.004 (2) | 0.004 (2) | -0.005 (2) |
| C31 | 0.0143 (18) | 0.021 (3) | 0.022 (4) | 0.0026 (15) | 0.000 (2) | -0.003 (3) |
| C32 | 0.022 (2) | 0.022 (3) | 0.028 (3) | 0.003 (2) | 0.001 (2) | -0.006 (2) |
| C33 | 0.028 (2) | 0.029 (3) | 0.035 (3) | 0.015 (2) | 0.007 (3) | -0.004 (3) |

| | | | | | | |
|-----|-----------|-----------|-----------|------------|------------|------------|
| C34 | 0.016 (2) | 0.042 (4) | 0.039 (4) | 0.004 (2) | 0.000 (2) | 0.002 (3) |
| C35 | 0.017 (2) | 0.044 (3) | 0.030 (3) | -0.006 (2) | -0.001 (2) | -0.010 (3) |
| C36 | 0.020 (3) | 0.022 (3) | 0.032 (4) | -0.003 (2) | 0.001 (3) | -0.005 (3) |

Geometric parameters (Å, °)

| | | | |
|---------------------------------------|-------------|--------------|-----------|
| Os1—C2 ⁱ | 2.169 (5) | C14—C15 | 1.386 (8) |
| Os1—C2 | 2.169 (5) | C14—H14A | 0.9500 |
| Os1—C1 | 2.195 (5) | C15—C16 | 1.387 (7) |
| Os1—C1 ⁱ | 2.195 (5) | C15—H15A | 0.9500 |
| Os1—C11 ⁱ | 2.4429 (12) | C16—H16A | 0.9500 |
| Os1—C11 | 2.4429 (12) | C21—C22 | 1.382 (6) |
| Os1—P1 | 2.5031 (12) | C21—C26 | 1.384 (7) |
| Os1—P1 ⁱ | 2.5031 (12) | C22—C23 | 1.387 (8) |
| P1—C11 | 1.836 (4) | C22—H22A | 0.9500 |
| P1—C31 | 1.848 (4) | C23—C24 | 1.364 (8) |
| P1—C21 | 1.858 (5) | C23—H23A | 0.9500 |
| C1—C2 | 1.404 (6) | C24—C25 | 1.379 (7) |
| C1—C4 ⁱ | 1.519 (7) | C24—H24A | 0.9500 |
| C1—H1A | 1.0000 | C25—C26 | 1.365 (7) |
| C2—C3 | 1.516 (7) | C25—H25A | 0.9500 |
| C2—H2A | 1.0000 | C26—H26A | 0.9500 |
| C3—C4 | 1.538 (7) | C31—C32 | 1.386 (7) |
| C3—H3A | 0.9900 | C31—C36 | 1.390 (7) |
| C3—H3B | 0.9900 | C32—C33 | 1.385 (7) |
| C4—C1 ⁱ | 1.519 (7) | C32—H32A | 0.9500 |
| C4—H4A | 0.9900 | C33—C34 | 1.381 (7) |
| C4—H4B | 0.9900 | C33—H33A | 0.9500 |
| C11—C12 | 1.392 (7) | C34—C35 | 1.368 (7) |
| C11—C16 | 1.400 (7) | C34—H34A | 0.9500 |
| C12—C13 | 1.375 (7) | C35—C36 | 1.383 (7) |
| C12—H12A | 0.9500 | C35—H35A | 0.9500 |
| C13—C14 | 1.380 (9) | C36—H36A | 0.9500 |
| C13—H13A | 0.9500 | | |
| C2 ⁱ —Os1—C2 | 94.3 (3) | C3—C4—H4B | 109.1 |
| C2 ⁱ —Os1—C1 | 78.95 (18) | H4A—C4—H4B | 107.8 |
| C2—Os1—C1 | 37.52 (16) | C12—C11—C16 | 118.9 (4) |
| C2 ⁱ —Os1—C1 ⁱ | 37.52 (16) | C12—C11—P1 | 121.8 (4) |
| C2—Os1—C1 ⁱ | 78.95 (18) | C16—C11—P1 | 118.9 (4) |
| C1—Os1—C1 ⁱ | 87.3 (3) | C13—C12—C11 | 121.7 (5) |
| C2 ⁱ —Os1—C11 ⁱ | 84.95 (14) | C13—C12—H12A | 119.2 |
| C2—Os1—C11 ⁱ | 158.22 (12) | C11—C12—H12A | 119.2 |
| C1—Os1—C11 ⁱ | 159.61 (12) | C12—C13—C14 | 118.6 (6) |
| C1 ⁱ —Os1—C11 ⁱ | 87.54 (13) | C12—C13—H13A | 120.7 |
| C2 ⁱ —Os1—C11 | 158.22 (12) | C14—C13—H13A | 120.7 |
| C2—Os1—C11 | 84.95 (14) | C13—C14—C15 | 121.4 (6) |

supplementary materials

| | | | |
|---------------------------------------|-------------|--------------|-----------|
| C1—Os1—C11 | 87.54 (13) | C13—C14—H14A | 119.3 |
| C1 ⁱ —Os1—C11 | 159.61 (12) | C15—C14—H14A | 119.3 |
| C11 ⁱ —Os1—C11 | 103.69 (6) | C14—C15—C16 | 119.7 (6) |
| C2 ⁱ —Os1—P1 | 82.14 (12) | C14—C15—H15A | 120.2 |
| C2—Os1—P1 | 120.55 (12) | C16—C15—H15A | 120.2 |
| C1—Os1—P1 | 84.48 (13) | C15—C16—C11 | 119.7 (6) |
| C1 ⁱ —Os1—P1 | 119.44 (12) | C15—C16—H16A | 120.1 |
| C11 ⁱ —Os1—P1 | 80.97 (4) | C11—C16—H16A | 120.1 |
| C11—Os1—P1 | 79.60 (4) | C22—C21—C26 | 117.9 (5) |
| C2 ⁱ —Os1—P1 ⁱ | 120.55 (12) | C22—C21—P1 | 117.2 (4) |
| C2—Os1—P1 ⁱ | 82.14 (12) | C26—C21—P1 | 124.4 (3) |
| C1—Os1—P1 ⁱ | 119.44 (12) | C21—C22—C23 | 121.0 (5) |
| C1 ⁱ —Os1—P1 ⁱ | 84.48 (13) | C21—C22—H22A | 119.5 |
| C11 ⁱ —Os1—P1 ⁱ | 79.60 (4) | C23—C22—H22A | 119.5 |
| C11—Os1—P1 ⁱ | 80.97 (4) | C24—C23—C22 | 120.1 (5) |
| P1—Os1—P1 ⁱ | 148.31 (6) | C24—C23—H23A | 120.0 |
| C11—P1—C31 | 104.8 (2) | C22—C23—H23A | 120.0 |
| C11—P1—C21 | 98.0 (2) | C23—C24—C25 | 119.3 (5) |
| C31—P1—C21 | 98.5 (2) | C23—C24—H24A | 120.4 |
| C11—P1—Os1 | 113.95 (14) | C25—C24—H24A | 120.4 |
| C31—P1—Os1 | 119.87 (15) | C26—C25—C24 | 120.8 (5) |
| C21—P1—Os1 | 118.45 (15) | C26—C25—H25A | 119.6 |
| C2—C1—C4 ⁱ | 120.5 (4) | C24—C25—H25A | 119.6 |
| C2—C1—Os1 | 70.2 (3) | C25—C26—C21 | 120.9 (4) |
| C4 ⁱ —C1—Os1 | 115.2 (3) | C25—C26—H26A | 119.5 |
| C2—C1—H1A | 114.5 | C21—C26—H26A | 119.5 |
| C4 ⁱ —C1—H1A | 114.5 | C32—C31—C36 | 117.5 (4) |
| Os1—C1—H1A | 114.5 | C32—C31—P1 | 119.0 (4) |
| C1—C2—C3 | 121.2 (4) | C36—C31—P1 | 123.3 (4) |
| C1—C2—Os1 | 72.3 (3) | C31—C32—C33 | 121.6 (5) |
| C3—C2—Os1 | 114.3 (3) | C31—C32—H32A | 119.2 |
| C1—C2—H2A | 114.2 | C33—C32—H32A | 119.2 |
| C3—C2—H2A | 114.2 | C34—C33—C32 | 119.8 (5) |
| Os1—C2—H2A | 114.2 | C34—C33—H33A | 120.1 |
| C2—C3—C4 | 112.7 (4) | C32—C33—H33A | 120.1 |
| C2—C3—H3A | 109.1 | C35—C34—C33 | 119.2 (5) |
| C4—C3—H3A | 109.1 | C35—C34—H34A | 120.4 |
| C2—C3—H3B | 109.1 | C33—C34—H34A | 120.4 |
| C4—C3—H3B | 109.1 | C34—C35—C36 | 120.9 (5) |
| H3A—C3—H3B | 107.8 | C34—C35—H35A | 119.5 |
| C1 ⁱ —C4—C3 | 112.7 (4) | C36—C35—H35A | 119.5 |
| C1 ⁱ —C4—H4A | 109.1 | C35—C36—C31 | 120.8 (5) |
| C3—C4—H4A | 109.1 | C35—C36—H36A | 119.6 |
| C1 ⁱ —C4—H4B | 109.1 | C31—C36—H36A | 119.6 |

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

Fig. 1

