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Asymmetric diosmium sawhorse complexes

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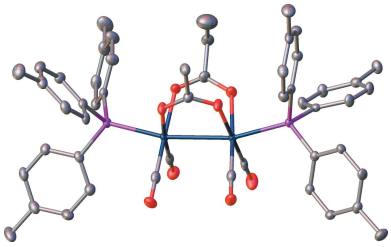
Three asymmetric diosmium(I) carbonyl sawhorse complexes have been prepared by microwave heating. One of these complexes is of the type $Os_2(\mu\text{-}O_2CR)(\mu\text{-}O_2CR')(CO)_4L_2$, with two different bridging carboxylate ligands, while the other two complexes are of the type $Os_2(\mu\text{-}O_2CR)_2(CO)_5L$, with one axial CO ligand and one axial phosphane ligand. The mixed carboxylate complex $Os_2(\mu\text{-acetate})(\mu\text{-propionate})(CO)_4[P(p\text{-tolyl})_3]_2$, (**1**), was prepared by heating $Os_3(CO)_{12}$ with a mixture of acetic and propionic acids, isolating $Os_2(\mu\text{-acetate})(\mu\text{-propionate})(CO)_6$, and then replacing two CO ligands with two phosphane ligands. This is the first example of an Os_2 sawhorse complex with two different carboxylate bridges. The syntheses of $Os_2(\mu\text{-acetate})_2(CO)_5[P(p\text{-tolyl})_3]$, (**3**), and $Os_2(\mu\text{-propionate})_2(CO)_5[P(p\text{-tolyl})_3]$, (**6**), involved the reaction of $Os_3(CO)_{12}$ with the appropriate carboxylic acid to initially produce $Os_2(\mu\text{-carboxylate})_2(CO)_6$, followed by treatment with refluxing tetrahydrofuran (THF) to form $Os_2(\mu\text{-carboxylate})_2(CO)_5(THF)$, and finally addition of tri-*p*-tolylphosphane to replace the THF ligand with the $P(p\text{-tolyl})_3$ ligand. Neutral complexes of the type $Os_2(\mu\text{-}O_2CR)_2(CO)_5L$ had not previously been subjected to X-ray crystallographic analysis. The more symmetrical disubstituted complexes, *i.e.* $Os_2(\mu\text{-formate})_2(CO)_4[P(p\text{-tolyl})_3]_2$, (**8**), $Os_2(\mu\text{-acetate})_2(CO)_4[P(p\text{-tolyl})_3]_2$, (**4**), and $Os_2(\mu\text{-propionate})_2(CO)_4[P(p\text{-tolyl})_3]_2$, (**7**), as well as the previously reported symmetrical unsubstituted complexes $Os_2(\mu\text{-acetate})_2(CO)_6$, (**2**), and $Os_2(\mu\text{-propionate})_2(CO)_6$, (**5**), were also prepared in order to examine the influence of axial ligand substitution on the Os—Os bond distance in these sawhorse molecules. Eight crystal structures have been determined and studied, namely μ-acetato-1κO:2$\kappa O'$-μ-propanoato-1κO:2$\kappa O'$-bis[tris(4-methylphenyl)phosphane]-1κP,2$\kappa P'$-bis(dicarbonylosmium)(*Os*—*Os*) dichloromethane monosolvate, $[Os_2(C_2H_3O_2)(C_3H_5O_2)(C_{21}H_{21}P)_2(CO)_4]\cdot CH_2Cl_2$, (**1**), bis($\mu$-acetato-1$\kappa O$:2$\kappa O'$)bis(tricarbonylosmium)(*Os*—*Os*), $[Os_2(C_2H_3O_2)_2(CO)_6]$, (**2**) (redetermined structure), bis(μ-acetato-1κO:2$\kappa O'$)pentacarbonyl-1$\kappa^2 C$,2$\kappa^3 C$-[tris(4-methylphenyl)phosphane-1κP]diosmium(*Os*—*Os*), $[Os_2(C_2H_3O_2)_2(C_{21}H_{21}P)(CO)_5]$, (**3**), bis($\mu$-acetato-1$\kappa O$:2$\kappa O'$)bis[tris(4-methylphenyl)phosphane]-1κP,2κP-bis(dicarbonylosmium)(*Os*—*Os*) *p*-xylene sesquisolvate, $[Os_2(C_2H_3O_2)_2(C_{21}H_{21}P)_2(CO)_4]\cdot 1.5C_8H_{10}$, (**4**), bis($\mu$-propanoato-1$\kappa O$:2$\kappa O'$)bis(tricarbonylosmium)(*Os*—*Os*), $[Os_2(C_3H_5O_2)_2(CO)_6]$, (**5**), pentacarbonyl-1$\kappa^2 C$,2$\kappa^3 C$-bis(μ-propanoato-1κO:2$\kappa O'$)-[tris(4-methylphenyl)phosphane-1κP]diosmium(*Os*—*Os*), $[Os_2(C_3H_5O_2)_2(C_{21}H_{21}P)(CO)_5]$, (**6**), bis($\mu$-propanoato-1$\kappa O$:2$\kappa O'$)bis[tris(4-methylphenyl)phosphane]-1κP,2κP-bis(dicarbonylosmium)(*Os*—*Os*) dichloromethane monosolvate, $[Os_2(C_3H_5O_2)_2(C_{21}H_{21}P)_2(CO)_4]\cdot CH_2Cl_2$, (**7**), and bis($\mu$-formato-1$\kappa O$:2$\kappa O'$)bis-[tris(4-methylphenyl)phosphane]-1κP,2κP-bis(dicarbonylosmium)(*Os*—*Os*), $[Os_2(CHO_2)_2(C_{21}H_{21}P)_2(CO)_4]$, (**8**).

1. Introduction

High-temperature reactions of $Os_3(CO)_{12}$ with carboxylic acids produce diosmium(I) sawhorse complexes, $Os_2(\mu$-



$O_2CR)_2(CO)_6$, with metal–metal single bonds (Crooks *et al.*, 1969). As shown in Scheme 1, the Os—Os vector forms the top of the sawhorse, while its legs consist of four equatorial CO ligands (Bullitt & Cotton, 1971). Two *cis*-carboxylate ligands are also bound in equatorial sites, and the remaining two CO ligands are positioned along the metal–metal axis. Johnson, Lewis, and co-workers first reported the preparation of these complexes in 1969, and they demonstrated that both axial CO ligands could be readily replaced by other ligands, including pyridine or tertiary phosphanes (Crooks *et al.*, 1969). In 1987, Deeming and co-workers showed that it is possible to replace only one of the axial CO ligands to produce asymmetric compounds of the type $Os_2(\mu\text{-}O_2CR)_2(CO)_5L$ (Deeming *et al.*, 1987). They refluxed a solution of $Os_2(\mu\text{-}O_2CR)_2(CO)_6$ in tetrahydrofuran (THF) to form $Os_2(\mu\text{-}O_2CR)_2(CO)_5(THF)$, and then replaced the THF ligand with a phosphane ligand (L). No crystal structures of neutral asymmetric Os_2 sawhorse complexes have been reported, but one anionic complex, $[Os_2(\mu\text{-}O_2CCH_3)_2Cl(CO)_5]^-$, containing one chloride ligand and one CO ligand in the axial positions, has been isolated and subjected to X-ray crystallographic analysis (Deeming *et al.*, 1987).

The use of microwave heating to prepare diosmium(I) sawhorses was first described in 2013 (Pyper *et al.*, 2013). A few new complexes were prepared and structurally characterized, and it was noted that metal–metal bond distances seemed to be correlated with the strength of the parent carboxylic acid. With increasing acid strength, the Os—Os bond distances get slightly longer. Since then, the microwave-assisted syntheses of several additional sawhorse complexes containing mono- and dicarboxylate ligands have been reported (Fikes *et al.*, 2014; Chor *et al.*, 2016; Gwini *et al.*, 2017). The goals of the current project were twofold: (i) to synthesize a sawhorse complex of the type $Os_2(\mu\text{-}O_2CR)(\mu\text{-}O_2CR')(CO)_6$ with two different bridging carboxylate ligands, and (ii) to perform single-crystal diffraction studies of at least one series of complexes with the formulae $Os_2(\mu\text{-}O_2CR)_2(CO)_6$, $Os_2(\mu\text{-}O_2CR)_2(CO)_5L$, and $Os_2(\mu\text{-}O_2CR)_2(CO)_4L_2$, in order to investigate the trend in Os—Os bond lengths. We now report that these goals have been met.

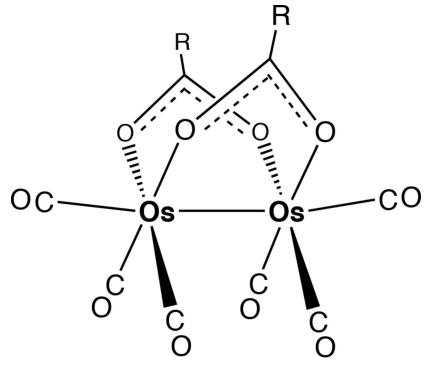
2. Experimental

2.1. Materials and methods

$Os_3(CO)_{12}$ and tri-*p*-tolylphosphane were purchased from Strem, while the other reagents were purchased from Sigma–Aldrich. All chemicals were used as received, and all operations were performed without taking precautions to exclude air and moisture. $Os_2(\mu$-acetate$)_2(CO)_6$, (**2**), $Os_2(\mu$-propionate$)_2(CO)_6$, (**5**), and $Os_2(\mu$-formate$)_2(CO)_6$, (**10**), were prepared according to published procedures (Pyper *et al.*, 2013). IR spectra were acquired using a Nicolet Avatar 320 FT–IR spectrometer with a CaF_2 solution cell. ^{1}H NMR spectra were recorded at 60 MHz on an Anasazi Eft-60 spectrometer. Preparative thin-layer chromatography (TLC) was carried out on Analtech silica gel 60 (0.50 mm) plates. Atlantic Microlab performed the elemental analyses.

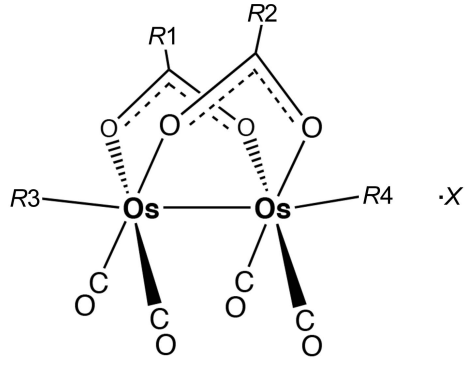
2.2. Synthesis and crystallization

2.2.1. Synthesis of (1). $Os_3(CO)_{12}$ (100.5 mg, 0.111 mmol), acetic acid (2.5 ml), and propionic acid (3.0 ml) were placed in a 35 ml glass reaction vessel and sealed with a polytetrafluoroethylene (PTFE)-lined cap. The mixture was stirred and irradiated in a CEM Discover-SP microwave reactor at 180 °C for 10 min. The excess acids were evaporated under a stream



Scheme 1

of compressed air, and the residue was redissolved in dichloromethane. TLC with an eluent of 7:3:1 heptane–dichloromethane–toluene gave four colorless bands, which were collected under UV light. Band 1 (R_F = 0.87) consisted of 10.9 mg (11.9% yield) of $Os_4(\mu\text{-H})_4(CO)_{12}$. IR ($\nu_{CO}$, $CHCl_3$): 2085 (*m*), 2067 (*vs*), 2020 (*s*), 1997 (*w*) cm^{-1}. Band 2 (R_F = 0.79) consisted of 29.4 mg (26.6% yield) of $Os_2(\mu$-acetate$)_2(CO)_6$. IR (ν_{CO}, $CHCl_3$): 2100 (*m*), 2067 (*vs*), 2015 (*s*), 1998 (*vs*) cm^{-1}. Band 3 (R_F = 0.74) consisted of 38.7 mg (34.2% yield) of $Os_2(\mu$-acetate$)(\mu$-propionate$)(CO)_6$. IR (ν_{CO}, $CHCl_3$): 2100 (*m*), 2066 (*vs*), 2014 (*s*), 1997 (*vs*) cm^{-1}. Band 4 (R_F = 0.67) consisted of 22.9 mg (19.9% yield) of



(**1**) *R*1 = Me, *R*2 = Et, *R*3/*R*4 = P(*p*-tolyl)$_3$, *X* = CH_2Cl_2
(**2**) *R*1/*R*2 = Me, *R*3/*R*4 = CO
(**3**) *R*1/*R*2 = Me, *R*3 = P(*p*-tolyl)$_3$, *R*4 = CO
(**4**) *R*1/*R*2 = Me, *R*3/*R*4 = P(*p*-tolyl)$_3$, *X* = 1.5C_4H_{10}
(**5**) *R*1/*R*2 = Et, *R*3/*R*4 = CO
(**6**) *R*1/*R*2 = Et, *R*3 = P(*p*-tolyl)$_3$, *R*4 = CO
(**7**) *R*1/*R*2 = Et, *R*3/*R*4 = P(*p*-tolyl)$_3$, *X* = CH_2Cl_2
(**8**) *R*1/*R*2 = H, *R*3/*R*4 = P(*p*-tolyl)$_3$

Scheme 2

$Os_2(\mu$-propionate$)_2(CO)_6$. IR (ν_{CO}, $CHCl_3$): 2100 (*m*), 2066 (*vs*), 2014 (*s*), 1997 (*vs*) cm^{-1}. The residue from band 3 was dissolved in 1,2-dichloroethane (25 ml) and acetonitrile

Table 1
Experimental details.

H-atom parameters were constrained.

	(**1**)	(**2**)	(**3**)	(**4**)
Crystal data				
Chemical formula	$[Os_2(C_2H_3O_2)(C_3H_5O_2)$-$(C_{21}H_{21}P)_2(CO)_4]$·-$CH_2Cl_2$	$[Os_2(C_2H_3O_2)_2(CO)_6]$	$[Os_2(C_2H_3O_2)_2(C_{21}H_{21}P)$-$(CO)_5]$	$[Os_2(C_2H_3O_2)_2(C_{21}H_{21}P)_2$-$(CO)_4]$·$1.5C_8H_{10}$
M_r	1318.17	666.55	942.88	1378.46
Crystal system, space group	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/n$	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$
Temperature (K)	100	200	100	293
a, b, c (Å)	18.5007 (4), 18.5396 (4), 14.8090 (3)	7.6949 (5), 14.3612 (10), 13.8623 (9)	10.452 (4), 11.229 (4), 13.045 (5)	12.81225 (16), 14.8455 (2), 16.6818 (2)
α, β, γ (°)	90, 91.9976 (17), 90	90, 105.202 (1), 90	82.813 (6), 89.157 (8), 89.457 (9)	98.1785 (11), 101.7904 (11), 113.3846 (14)
V (Å^3)	5076.35 (18)	1478.29 (17)	1518.8 (10)	2761.37 (7)
Z	4	4	2	2
Radiation type	Cu $K\alpha$	Mo $K\alpha$	Mo $K\alpha$	Cu $K\alpha$
μ (mm^{-1})	11.30	17.22	8.46	9.55
Crystal size (mm)	0.22 × 0.06 × 0.03	0.19 × 0.12 × 0.07	0.27 × 0.14 × 0.12	0.11 × 0.07 × 0.02
Data collection				
Diffractometer	Rigaku SuperNova AtlasS2 CCD	Bruker APEXII CCD	Rigaku SCX-Mini Mercury 2+ CCD	Rigaku SuperNova AtlasS2 CCD
Absorption correction	Gaussian (*CrysAlis PRO*; Rigaku OD, 2017)	Multi-scan (*SADABS*; Bruker, 2008)	Multi-scan (*ABSCOR*; Higashi, 2001)	Gaussian (*CrysAlis PRO*; Rigaku OD, 2017)
T_{min}, T_{max}	0.428, 1.000	0.295, 0.747	0.605, 1.00	0.626, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	20129, 9265, 8475	14002, 3252, 3003	32350, 6915, 6534	49090, 9768, 8691
R_{int}	0.024	0.046	0.032	0.066
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.602	0.641	0.648	0.595
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.023, 0.055, 1.00	0.023, 0.055, 1.09	0.020, 0.045, 1.02	0.027, 0.068, 1.05
No. of reflections	9265	3252	6915	9768
No. of parameters	632	202	385	678
No. of restraints	112	0	0	0
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.16, −1.05	1.80, −1.33	2.80, −1.05	2.09, −1.11

	(**5**)	(**6**)	(**7**)	(**8**)
Crystal data				
Chemical formula	$[Os_2(C_3H_5O_2)_2(CO)_6]$	$[Os_2(C_3H_5O_2)_2(C_{21}H_{21}P)$-$(CO)_5]$	$[Os_2(C_3H_5O_2)_2(C_{21}H_{21}P)_2$-$(CO)_4]$·$CH_2Cl_2$	$[Os_2(CHO_2)_2(C_{21}H_{21}P)_2$-$(CO)_4]$
M_r	694.60	970.94	1332.20	1191.17
Crystal system, space group	Orthorhombic, *Pbca*	Triclinic, $P\overline{1}$	Monoclinic, $P2_1/c$	Triclinic, $P\overline{1}$
Temperature (K)	100	100	100	200
a, b, c (Å)	9.79942 (10), 15.51827 (19), 21.7339 (2)	10.0952 (2), 11.6630 (3), 13.7536 (3)	18.4968 (6), 18.7369 (7), 14.9214 (6)	10.7707 (4), 10.8014 (4), 20.3790 (7)
α, β, γ (°)	90, 90, 90	88.6185 (19), 86.7627 (18), 86.9563 (19)	90, 91.806 (2), 90	104.393 (1), 91.009 (1), 98.993 (1)
V (Å^3)	3305.08 (6)	1614.12 (6)	5168.8 (3)	2264.27 (14)
Z	8	2	4	2
Radiation type	Cu $K\alpha$	Cu $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	29.08	15.55	5.13	5.73
Crystal size (mm)	0.08 × 0.06 × 0.04	0.21 × 0.14 × 0.06	0.28 × 0.12 × 0.09	0.15 × 0.11 × 0.04
Data collection				
Diffractometer	Rigaku SuperNova AtlasS2 CCD	Rigaku SuperNova AtlasS2 CCD	Rigaku SCX-Mini Mercury 2+ CCD	Bruker APEXII CCD
Absorption correction	Multi-scan *CrysAlis PRO* (Rigaku OD, 2015)	Multi-scan *CrysAlis PRO* (Rigaku OD, 2015)	Multi-scan (*ABSCOR*; Higashi, 2001)	Multi-scan (*SADABS*; Bruker, 2008)
T_{min}, T_{max}	0.637, 1.000	0.389, 1.000	0.673, 1.00	0.538, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	16571, 3300, 3196	30757, 6461, 6196	54214, 11794, 9639	30910, 9960, 8554
R_{int}	0.027	0.038	0.060	0.058
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.622	0.622	0.649	0.641
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.018, 0.039, 1.03	0.019, 0.046, 1.08	0.036, 0.071, 1.07	0.030, 0.082, 1.06
No. of reflections	3300	6461	11794	9960

Table 1 (continued)

	(5)	(6)	(7)	(8)
No. of parameters	219	402	624	548
No. of restraints	0	0	20	0
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.55, −0.93	0.55, −1.21	1.39, −1.32	1.19, −1.09

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015, 2017), *APEX2* (Bruker, 2009), *CrystalClear* (Rigaku, 2008), *SAINT* (Bruker, 2009), *SHELXT* (Sheldrick, 2015*a*), *SIR97* (Altomare *et al.*, 1999), *SHELXL2018* (Sheldrick, 2015*b*), *SHELXL2013* (Sheldrick, 2015*b*), *OLEX2* (Dolomanov *et al.*, 2009), and *XP* in *SHELXTL*/*PC* (Sheldrick, 1990).

(5 ml). Tri-*p*-tolylphosphane (0.0260 g, 0.0854 mmol) was added and the mixture was refluxed for 50 min. The solvent was removed and the residue was dissolved in CH_2Cl_2. TLC with an eluent of 3:2 hexanes–dichloromethane gave two major bands. Band 1 consisted of 9.7 mg of unreacted $Os_2(\mu$-acetate$)(\mu$-propionate$)(CO)_6$. Band 2 consisted of 36.8 mg of (**1**) [18.0% yield based on $Os_3(CO)_{12}$]. IR (ν_{CO}, $CHCl_3$): 2011 (*vs*), 1967 (*m*), 1934 (*vs*) cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 7.30 (*m*, 24H), 2.35 (*s*, 18H), 1.88 (*q*, 2H), 1.59 (*s*, 3H), 0.58 (*t*, 3H). Analysis calculated (%) for $C_{51}H_{50}O_8Os_2P_2$: C 49.67, H 4.09; found: C 49.78, H 4.04. Single crystals suitable for X-ray diffraction were obtained by slow diffusion of *n*-hexane into a dichloromethane solution at ambient temperature.

2.2.2. Synthesis of (3). A mixture of $Os_3(CO)_{12}$ (80.2 mg, 0.0884 mmol) and acetic acid (6 ml) was stirred and irradiated in a microwave reactor at 180 °C for 10 min. The excess acid was evaporated under a stream of air and the residue was redissolved in tetrahydrofuran (25 ml). This solution was refluxed for 2.5 h with vigorous stirring and then cooled to 0 °C in an ice bath. A second solution consisting of tri-*p*-tolylphosphane (30.4 mg, 0.0999 mmol) dissolved in THF (20 ml) was added dropwise over a period of 1 h to the solution in the ice bath. The resulting mixture was allowed to warm slowly to room temperature and the solvent was then removed by rotary evaporation. The residue was dissolved in CH_2Cl_2 and subjected to TLC with an eluent of 1:1 hexanes–CH_2Cl_2. Four bands were collected. Band 1 (R_F = 0.90) consisted of 3.7 mg (5.1% yield) of $Os_4(\mu$-H$)_4(CO)_{12}$. Band 2 (R_F = 0.54) consisted of 14.2 mg (16.1% yield) of $Os_2(\mu$-acetate$)_2(CO)_6$, (**2**). Band 3 (R_F = 0.43) consisted of 10.8 mg (6.7% yield) of $Os_2(\mu$-acetate$)_2(CO)_4[P(p$-tolyl$)_3]_2$, (**4**). IR (ν_{CO}, $CHCl_3$): 2011 (*vs*), 1967 (*m*), 1935 (*vs*) cm^{-1}. Band 4 (R_F = 0.35) consisted of 34.3 mg (27.4% yield) of $Os_2(\mu$-acetate$)_2(CO)_5[P(p$-tolyl$)_3]$, (**3**). IR (ν_{CO}, $CHCl_3$): 2072 (*vs*), 2001 (*vs*), 1977 (*s*), 1923 (*m*) cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 7.39 (*m*, 12H), 2.40 (*m*, 9H), 1.54 (*s*, 6H). Analysis calculated (%) for $C_{30}H_{27}O_9Os_2P$: C 38.21, H 2.89; found: C 38.57, H 2.99. Single crystals were obtained by slow diffusion of *n*-hexane into a dichloromethane solution at ambient temperature.

2.2.3. Synthesis of (4). A mixture of $Os_3(CO)_{12}$ (49.4 mg, 0.0545 mmol) and acetic acid (7 ml) was stirred and irradiated in a microwave reactor at 185 °C for 10 min. The excess acid was evaporated under a stream of air and the residue was redissolved in a mixture of chloroform (25 ml) and acetonitrile (3 ml). Tri-*p*-tolylphosphane (81.7 mg, 0.268 mmol) was added and the solution refluxed for 55 min. The solvent was removed and the residue dissolved in CH_2Cl_2. Three bands were collected after TLC with an eluent of 1.25:1 hexanes–CH_2Cl_2. The first and second bands contained 2.7 mg of $Os_4(\mu$-H$)_4(CO)_{12}$ and 7.1 mg of (**2**), respectively. Band 3 consisted of 54.0 mg (54.1% yield) of (**4**). IR (ν_{CO}, $CHCl_3$): 2011 (*vs*), 1967 (*w*), 1934 (*vs*) cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 7.35 (*m*, 24H), 2.36 (*m*, 18H), 1.60 (*t*, 6H). Analysis calculated (%) for $C_{50}H_{48}O_8Os_2P_2$: C 49.25, H 3.97; found: C 50.02, H 4.08. Single crystals were obtained by slow evaporation of a *p*-xylene solution at ambient temperature.

2.2.4. Synthesis of (6). The procedure described above for the synthesis of (**3**) was employed. $Os_3(CO)_{12}$ (80.6 mg, 0.0889 mmol), propionic acid (6.5 ml), and tri-*p*-tolylphosphane (39.3 mg, 0.129 mmol) were used. TLC with an eluent of 1:1 hexanes–CH_2Cl_2 produced three bands. Band 1 consisted of 13.1 mg (17.8% yield) of $Os_4(\mu$-H$)_4(CO)_{12}$. Band 2 consisted of 3.6 mg (2.2% yield) of $Os_2(\mu$-propionate$)_2(CO)_4[P(p$-tolyl$)_3]_2$, (**7**). Band 3 consisted of 57.8 mg (44.6% yield) of $Os_2(\mu$-propionate$)_2(CO)_5[P(p$-tolyl$)_3]$, (**6**). IR (ν_{CO}, $CHCl_3$): 2072 (*vs*), 2002 (*vs*), 1976 (*s*), 1923 (*m*) cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 7.40 (*m*, 12H), 2.39 (*m*, 9H), 2.05 (*q*, 4H), 0.86 (*t*, 6H). Analysis calculated (%) for $C_{32}H_{31}O_9Os_2P$: C 39.58, H 3.22; found: C 39.97, H 3.36. Single crystals were obtained by slow diffusion of *n*-hexane into a dichloromethane solution at ambient temperature.

2.2.5. Synthesis of (7). The procedure described above for the synthesis of (**4**) was employed. $Os_3(CO)_{12}$ (78.4 mg, 0.0865 mmol), propionic acid (8 ml), and tri-*p*-tolylphosphane (123.7 mg, 0.406 mmol) were used. Two TLC bands were collected. Band 1 consisted of 7.4 mg of $Os_4(\mu$-H$)_4(CO)_{12}$. Band 2 consisted of 89.9 mg (55.5% yield) of $Os_2(\mu$-propionate$)_2(CO)_4[P(p$-tolyl$)_3]_2$, (**7**). IR (ν_{CO}, $CHCl_3$): 2011 (*vs*), 1967 (*w*), 1934 (*vs*) cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 7.36 (*m*, 24H), 2.36 (*m*, 18H), 1.83 (*q*, 4H), 0.59 (*t*, 6H). Analysis calculated (%) for $C_{52}H_{52}O_8Os_2P_2$: C 50.07, H 4.20; found: C 50.02, H 4.18. Single crystals were obtained by slow diffusion of *n*-hexane into a dichloromethane solution at ambient temperature.

2.2.6. Synthesis of (8). $Os_2(\mu$-formate$)_2(CO)_6$ (59.6 mg, 0.0933 mmol) and tri-*p*-tolylphosphane (142 mg, 0.467 mmol) were dissolved in a mixture of chloroform (25 ml) and acetonitrile (3 ml). The solution was refluxed for 1 h. The solvent was removed and the residue was dissolved in CH_2Cl_2. TLC with an eluent of 1:1 hexanes–CH_2Cl_2 produced one major band, which consisted of 73.8 mg (66.4% yield) of $Os_2(\mu$-formate$)_2(CO)_4[P(p$-tolyl$)_3]_2$, (**8**). IR (ν_{CO}, $CHCl_3$): 2012 (*vs*), 1969 (*w*), 1938 (*vs*) cm^{-1}. Analysis calculated (%) for $C_{48}H_{44}O_8Os_2P_2$: C 48.40, H 3.72; found: C 47.36, H 3.65%. Single crystals were obtained by slow evaporation of a concentrated dichloromethane solution at ambient temperature.

2.2.7. Crystallization of (5). Single crystals of (**2**) were obtained by slow diffusion of *n*-hexane into a dichloromethane solution at ambient temperature. Single crystals of (**5**) were obtained by slow diffusion of *n*-pentane into a chloroform solution at ambient temperature.

2.3. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. All H atoms were placed in calculated positions and refined using a riding model. For (**1**), both bridging carboxylate positions encompassed acetate and propionate moieties disordered in 50:50 ratios with respect to one another. Bond distances between the carboxylate C atoms and the methyl and ethyl groups were restrained to idealized values, and the anisotropic displacement parameters of the acetate and propionate C and O atoms were restrained. For (**7**), the methyl group of one propionate ligand is disordered so that there are two alternative orientations for that terminal C atom (C45 and C45*A*). The occupancy ratio refined to 0.726 (13):0.274 (13). The geometry of the two components was restrained to be equivalent, atoms C45 and C45*A* were restrained to approximate isotropic behavior, and similar anisotropic displacement parameters were used for both atoms.

3. Results and discussion

3.1. Synthesis and spectroscopy

A combination of $Os_3(CO)_{12}$, acetic acid, and propionic acid was used to successfully synthesize the first example of a mixed carboxylate sawhorse complex, namely $Os_2(\mu$-acetate)-$(\mu$-propionate$)(CO)_6$, which was sandwiched between $Os_2(\mu$-acetate$)_2(CO)_6$ and $Os_2(\mu$-propionate$)_2(CO)_6$ on the preparative TLC plates. We have observed that the latter two compounds decompose slowly over a period of several weeks. However, upon replacing the two axial CO ligands in these compounds with phosphane ligands, the resulting complexes are stable for months or years. Thus, we chose to immediately convert the asymmetric product $Os_2(\mu$-acetate$)(\mu$-propionate$)(CO)_6$ into $Os_2(\mu$-acetate$)(\mu$-propionate$)(CO)_4[P(p$-tolyl$)_3]_2$, (**1**), before conducting further studies. As expected, the overall yield was low at 18%. Two three-membered series of $Os_2(\mu$-$O_2CR)_2(CO)_{6-n}L_n$ complexes, in which *L* is tri-*p*-tolylphosphane and n = 0, 1, or 2, were prepared by similar methods. One such series consists of complexes (**2**), (**3**), and (**4**), for which *R* = Me (acetate) and n = 0, 1, and 2, respectively. The other series consists of complexes (**5**), (**6**), and (**7**), for which *R* = Et (propionate) and n = 0, 1, and 2, respectively. The yields for complexes (**2**), (**5**), and (**10**) with no phosphane ligands (n = 0) were 90% or greater (Pyper *et al.*, 2013). The yields for the three disubstituted complexes (**4**), (**7**), and (**8**), with n = 2, were between 54 and 66%. The yields for the two

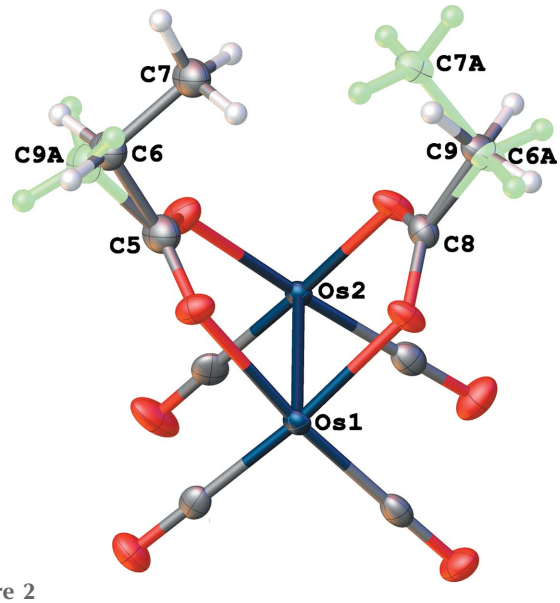


Figure 2
The structure of the core portion of complex (**1**), showing the 50:50 disorder of the bridging acetate and propionate ligands. The phosphane ligands have been omitted for clarity.

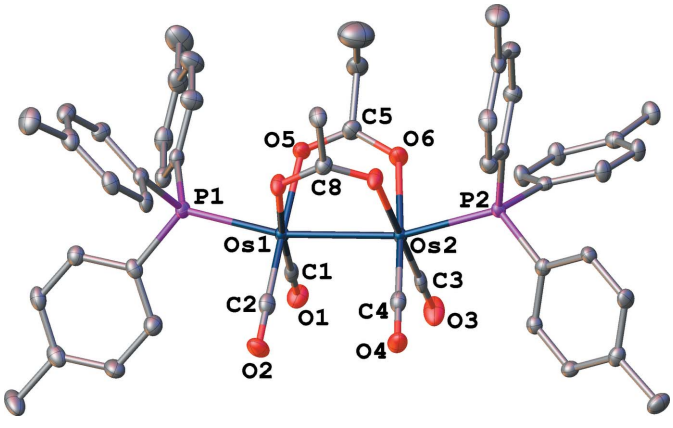


Figure 1
The molecular structure of the metal complex in (**1**). H atoms and solvent molecules have been omitted for clarity. Displacement ellipsoids are drawn at the 50% probability level.

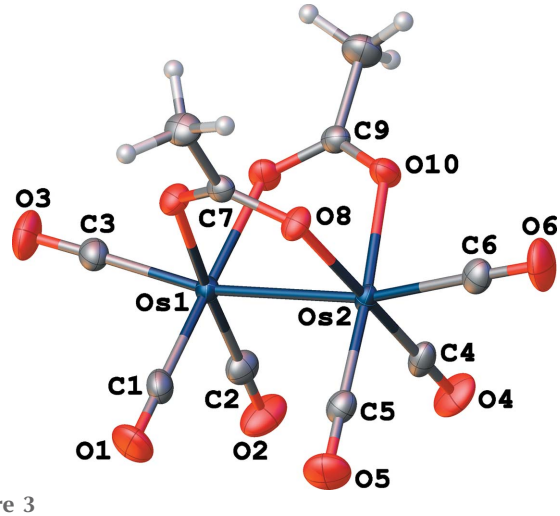


Figure 3
The molecular structure of (**2**), with displacement ellipsoids drawn at the 50% probability level.

Table 2
Carbonyl stretching frequencies of the diosmium sawhorse complexes (**1**)–(**10**) of the type $Os_2(O_2CR)_2(CO)_{6-n}L_n$ for which n = 0, 1, or 2, and L is tri-*p*-tolylphosphane.

Complex	ν_{CO} (cm^{-1})
Os_2(acetate)$_2$(CO)$_6$, (**2**)	2100 (*m*), 2067 (*vs*), 2015 (*s*), 1998 (*vs*)
Os_2(propionate)$_2$(CO)$_6$, (**5**)	2099 (*m*), 2066 (*vs*), 2014 (*s*), 1997 (*vs*)
Os_2(benzoate)$_2$(CO)$_6$, (**9**)	2099 (*m*), 2066 (*vs*), 2015 (*s*), 1998 (*vs*)
Os_2(formate)$_2$(CO)$_6$, (**10**)	2104 (*m*), 2071 (*vs*), 2021 (*s*), 2004 (*vs*)
Os_2(acetate)$_2$(CO)$_5$[*P*(*p*-tolyl)$_3$], (**3**)	2072 (*vs*), 2001 (*vs*), 1977 (*s*), 1923 (*m*)
Os_2(propionate)$_2$(CO)$_5$[*P*(*p*-tolyl)$_3$], (**6**)	2072 (*vs*), 2002 (*vs*), 1976 (*s*), 1923 (*m*)
Os_2(acetate)(propionate)(CO)$_4$[*P*(*p*-tolyl)$_3$]$_2$, (**1**)	2011 (*vs*), 1967 (*m*), 1934 (*vs*)
Os_2(acetate)$_2$(CO)$_4$[*P*(*p*-tolyl)$_3$]$_2$, (**4**)	2011 (*vs*), 1967 (*m*), 1934 (*vs*)
Os_2(propionate)$_2$(CO)$_4$[*P*(*p*-tolyl)$_3$]$_2$, (**7**)	2011 (*vs*), 1967 (*m*), 1934 (*vs*)
Os_2(formate)$_2$(CO)$_4$[*P*(*p*-tolyl)$_3$]$_2$, (**8**)	2017 (*vs*), 1972 (*m*), 1941 (*vs*)

asymmetric complexes (**3**) and (**6**), with n = 1, were clearly the lowest at 27 and 45%, respectively.

All of the complexes display characteristic IR carbonyl stretching frequencies in the range 1900–2110 cm^{-1}. The ν_{CO} frequencies of complexes (**1**)–(**10**) are reported in Table 2. The degree of phosphane substitution is easily determined by examination of the IR spectra. Complexes with no phosphane ligands (n = 0) display four absorption maxima with an *m*/*vs*/*s*/*vs* pattern. Complexes with one phosphane ligand (n = 1) show four absorption maxima, but the pattern is *vs*/*vs*/*s*/*m*. Complexes with two phosphane ligands (n = 2) display only three absorption maxima with a *vs*/*m*/*vs* pattern. The ν_{CO} frequencies for the formate complexes are higher than those containing the other carboxylate ligands. This is most likely due to the fact that formate is the weakest base among these ligands, which results in a lesser amount of Os—CO π-back-bonding and comparatively stronger C—O bonds in the formate complexes.

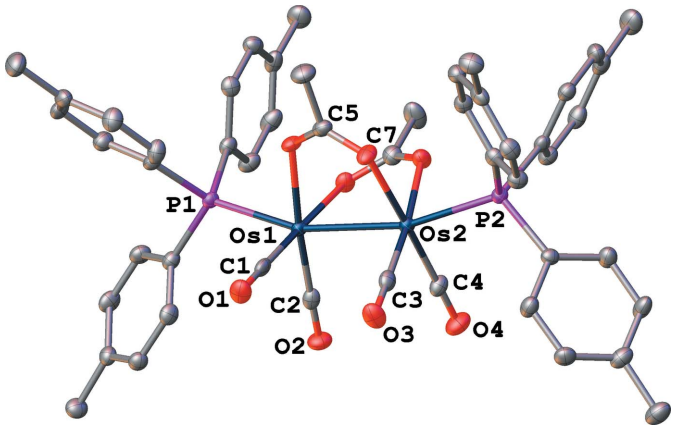


Figure 5
The molecular structure of (**4**), with displacement ellipsoids drawn at the 50% probability level. H atoms and solvent molecules have been omitted for clarity.

3.2. X-ray crystallography

X-ray data were collected at 100 K for five of the new complexes [*i.e.* (**1**), (**3**), (**5**), (**6**), and (**7**)], at 200 K for two [*i.e.* (**2**) and (**8**)], and at 293 K for complex (**4**). The data collection temperature for previously reported complexes (**9**) and (**10**) was 100 K. Using the same temperature for all of the diffraction experiments would have been ideal for the purposes of comparing the structural details, however, the bond lengths and angles associated with the Os atoms at the

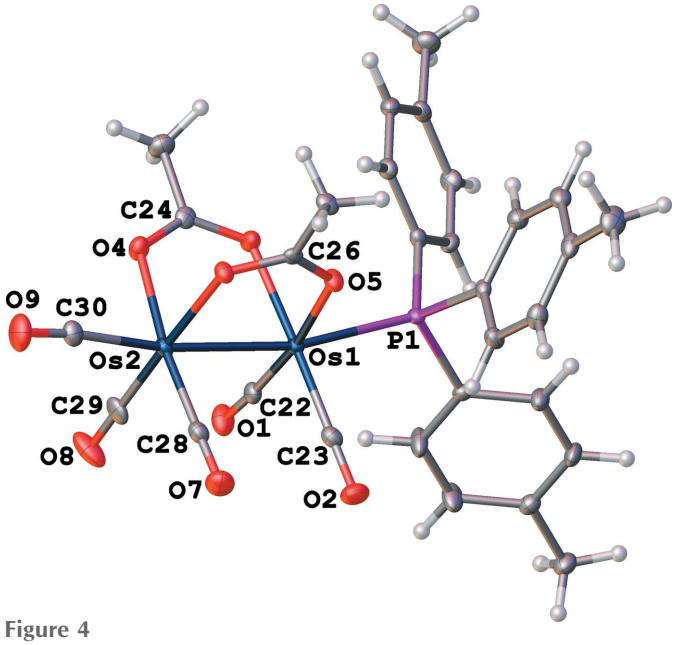


Figure 4
The molecular structure of (**3**), with displacement ellipsoids drawn at the 50% probability level.

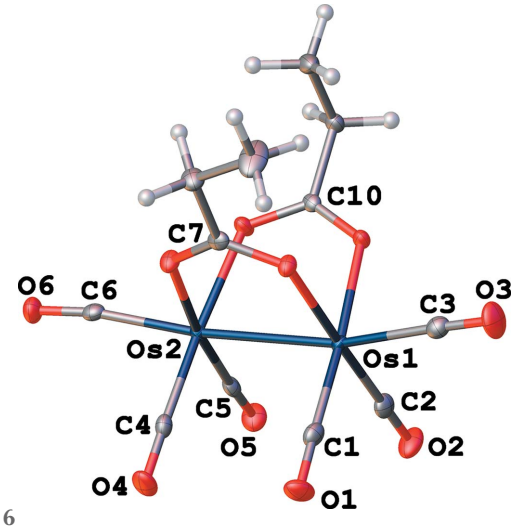


Figure 6
The molecular structure of (**5**), with displacement ellipsoids drawn at the 50% probability level.

Table 3
Os—Os bond lengths (Å) for eight $Os_2(O_2CR)_2(CO)_{6-n}L_n$ sawhorse complexes (L is tri-*p*-tolylphosphane).

Carboxylate group	n = 0	n = 1	n = 2
Formate	2.7545 (2)	–	2.7388 (2)
Acetate	2.7419 (3)	2.7624 (8)	2.7534 (2)
Propionate	2.7523 (2)	2.7479 (2)	2.7677 (3)

core of these complexes are unlikely to vary significantly over this temperature range.

The complexes reported herein all have the typical sawhorse molecular geometry, with two *cis*-carboxylate ligands, four equatorial CO ligands, two axial CO or phosphane ligands, and a metal–metal single bond. The molecular structure of the metal complex in (**1**) is illustrated in Fig. 1. Not surprisingly, there is disorder associated with the methyl group of the acetate ligand and the ethyl group of the propionate ligand such that each appears at both carboxylate positions 50% of the time. The disordered ligands are shown in Fig. 2. The molecular geometries of (**2**), (**3**), and (**4**) are illustrated in Figs. 3, 4, and 5, respectively. A crystal structure of (**2**) was reported in 1971 (Bullitt & Cotton, 1971), but we chose to repeat this analysis in order to obtain more precise structural details. The molecular structures of (**5**), (**6**), and (**7**) are illustrated in Figs. 6, 7, and 8, respectively. The complexes $Os_2(\mu$-formate$)_2(CO)_6$, (**10**), and $Os_2(\mu$-formate$)_2(CO)_4[P(p$-tolyl$)_3]_2$, (**8**), were also included in this study. The crystal structure of (**10**) was reported several years ago (Pyper *et al.*, 2013). The molecular structure of (**8**) is illustrated in Fig. 9.

We assumed that the Os—Os bond distances in each series would steadily change with an increasing number of phosphane ligands. Clearly, that was not the case. The Os—Os bond distances for (**2**)–(**8**) and $Os_2(\mu$-formate$)_2(CO)_6$ are listed in Table 3. The values range from 2.7388 (2) Å for (**8**) to 2.7677 (3) Å for (**7**). Replacing one axial carbonyl ligand with one phosphane ligand resulted in an increase in the Os—Os bond distance for the acetate series [complexes (**2**) and (**3**)], but a decrease in the Os—Os distance for the propionate series [complexes (**5**) and (**6**)]. Replacing a second axial CO ligand with another phosphane ligand resulted in a decrease in the Os—Os bond distance for the acetate series [(**3**) and (**4**)], but an increase in the Os—Os distance for the propionate series [(**6**) and (**7**)]. There was an overall increase in the Os—Os distance in both the acetate and propionate complexes when two CO ligands were replaced by two phosphane ligands, but this distance decreased in the formate complexes. No logical pattern could be deduced.

As mentioned above, it was suggested previously that the Os—Os bond distances in diosmium(I) carbonyl sawhorse complexes are correlated with the strength of the parent carboxylic acid (Pyper *et al.*, 2013). Metal–metal distances became slightly longer as the acid strength increased (or as the strength of the conjugate base decreased). At that time, however, only three such sawhorse complexes had been subjected to X-ray crystallographic analysis, namely $Os_2(\mu$-acetate$)_2(CO)_6$, (**2**), $Os_2(\mu$-benzoate$)_2(CO)_6$, (**9**), and $Os_2(\mu$-formate$)_2(CO)_6$, (**10**). In addition, the 1971 crystal structure of

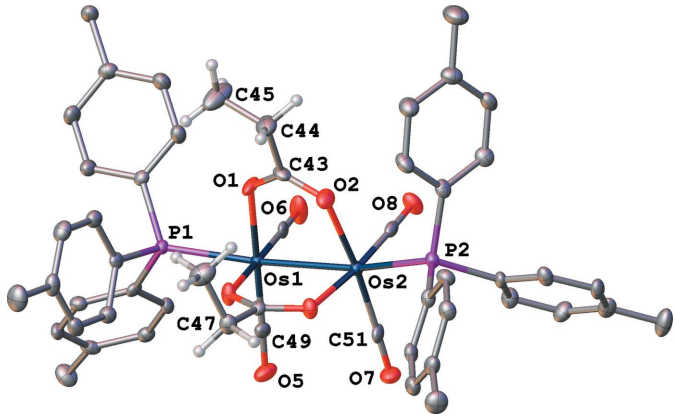


Figure 8
The molecular structure of (**7**), with displacement ellipsoids drawn at the 50% probability level. Tolyl H atoms and solvent molecules have been omitted for clarity.

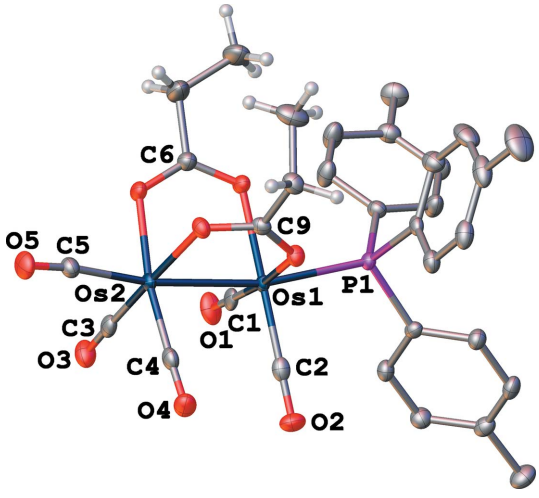


Figure 7
The molecular structure of (**6**), with displacement ellipsoids drawn at the 50% probability level. All tolyl H atoms have been omitted for clarity.

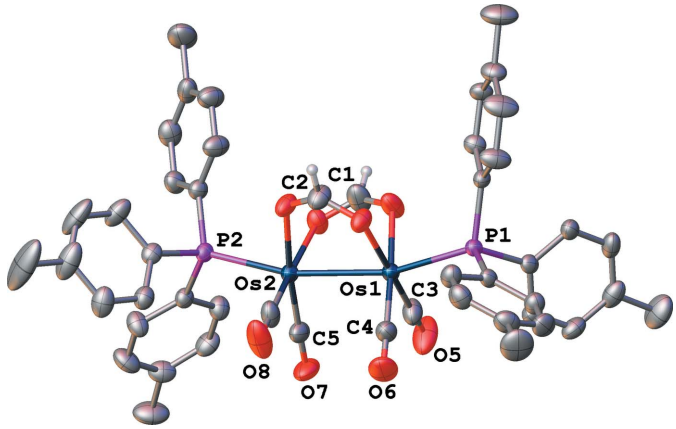


Figure 9
The molecular structure of (**8**), with displacement ellipsoids drawn at the 50% probability level. All tolyl H atoms have been omitted for clarity.

Table 4
Os—Os bond lengths (Å) and parent acid pK_a values for four $Os_2(O_2CR)_2(CO)_6$ complexes.

Compound	Acid pK_a	Os—Os
Os_2(formate)$_2$(CO)$_6$, (**10**)	3.75	2.7545 (2)
Os_2(benzoate)$_2$(CO)$_6$, (**9A**)	4.19	2.7495 (3)
Os_2(benzoate)$_2$(CO)$_6$, (**9B**)	4.19	2.7470 (3)
Os_2(acetate)$_2$(CO)$_6$, (**2**)	4.76	2.7419 (3)
Os_2(propionate)$_2$(CO)$_6$, (**5**)	4.88	2.7523 (2)

the acetate complex was somewhat rudimentary by present day standards. After obtaining the crystal structure of $Os_2(\mu$-propionate$)_2(CO)_6$ and redetermining the structure of $Os_2(\mu$-acetate$)_2(CO)_6$, we can now report that there is no apparent correlation between the Os—Os bond distance and the basicity of the carboxylate ligands. Table 4 lists the Os—Os bond distance along with the parent acid pK_a for the $Os_2(\mu$-$O_2CR)_2(CO)_6$ complexes, in which R is H, Me, Et, and Ph. The two complexes containing the carboxylate groups with the highest and lowest basicity, *i.e.* propionate and formate, respectively, have the two longest Os—Os bonds, while the acetate complex (with the second highest carboxylate basicity) has the shortest Os—Os bond. Neither the basicity of the carboxylate ligands nor the number of phosphane ligands have a predictable influence on the metal–metal bond length.

There are two independent molecules per unit cell in the previously reported crystal structure of $Os_2(\mu$-benzoate$)_2$-$(CO)_6$, (**9**) (Pyper *et al.*, 2013). One of these molecules, *i.e* (**9A**), has an Os—Os bond distance of 2.7495 (3) Å, while the other, *i.e* (**9B**), has an Os—Os bond distance of 2.7470 (3) Å. These molecules also have significantly different average C_{eq}—Os—Os—C_{eq} (where C_{eq} is an equatorial carbonyl C atom) torsion angles. The longer Os—Os bond length is associated with the molecule that has the smaller torsion angle. We wondered whether this might be a general trend, so we plotted Os—Os bond distance *versus* the average C_{eq}—Os—Os—C_{eq} torsion angle for the complexes in this study. We

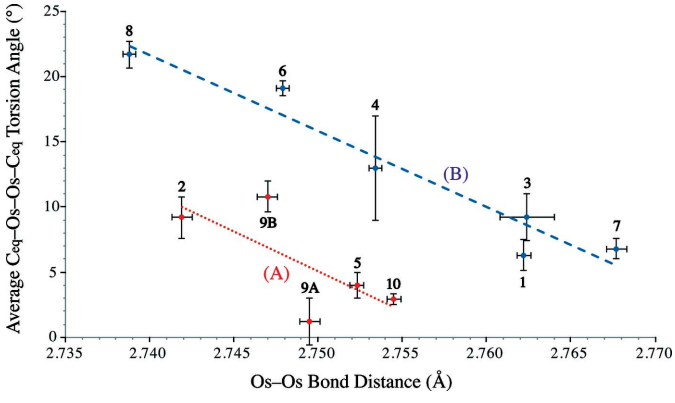


Figure 10
Plots of average C_{eq}—Os—Os—C_{eq} torsion angle *versus* Os—Os bond distance for (**A**) five $Os_2(O_2CR)_2(CO)_6$ sawhorse molecules and (**B**) six $Os_2(O_2CR)_2(CO)_{6-n}L_n$ sawhorse molecules. Data points are numbered according to the list of complexes in Table 5. Error bars represent twice the s.u. values for both distances and angles.

Table 5
Os—Os bond lengths (Å) and average C_{eq}—Os—Os—C_{eq} torsion angles (°) for 11 $Os_2(O_2CR)_2(CO)_{6-n}L_n$ sawhorse molecules (L is tri-*p*-tolylphosphane).

Compound	Bond length	Average torsion angle
Without phosphane ligands		
Os_2(acetate)$_2$(CO)$_6$, (**2**)	2.7419 (3)	9.2 (8)
Os_2(propionate)$_2$(CO)$_6$, (**5**)	2.7523 (2)	4.0 (5)
Os_2(benzoate)$_2$(CO)$_6$, (**9A**)	2.7495 (3)	1.2 (9)
Os_2(benzoate)$_2$(CO)$_6$, (**9B**)	2.7470 (3)	10.8 (6)
Os_2(formate)$_2$(CO)$_6$, (**10**)	2.7545 (2)	2.9 (3)
With phosphane ligands		
Os_2(acetate)(propionate)(CO)$_4$[P(*p*-tolyl)$_3$]$_2$, (**1**)	2.7623 (2)	6.3 (6)
Os_2(acetate)$_2$(CO)$_5$[P(*p*-tolyl)$_3$], (**3**)	2.7624 (8)	9.2 (9)
Os_2(acetate)$_2$(CO)$_4$[P(*p*-tolyl)$_3$]$_2$, (**4**)	2.7534 (2)	13 (3)
Os_2(propionate)$_2$(CO)$_5$[P(*p*-tolyl)$_3$], (**6**)	2.7479 (2)	19.1 (3)
Os_2(propionate)$_2$(CO)$_4$[P(*p*-tolyl)$_3$]$_2$, (**7**)	2.7677 (3)	6.8 (4)
Os_2(formate)$_2$(CO)$_4$[P(*p*-tolyl)$_3$]$_2$, (**8**)	2.7388 (2)	21.7 (5)

assumed that crystal packing forces are primarily responsible for the torsional twists in these complexes, and we thought the presence of relatively bulky phosphane ligands would have a significantly different effect on the arrangement of molecules in the crystal lattice *versus* the absence of such ligands. Thus, we divided the complexes into two categories: (i) those containing one or two axial phosphane ligands and (ii) those with only CO ligands in axial positions. The data are listed in Table 5. A plot of average C_{eq}—Os—Os—C_{eq} torsion angle *versus* Os—Os bond length with two different trend lines, one for the five molecules with no phosphane ligands and another for the six molecules with phosphane ligands, is given in Fig. 10. For both series, there is a steady decrease in the Os—Os bond length as the average torsion angle increases. The slopes of the two trend lines differ by less than 5%.

4. Conclusions

Three new asymmetric diosmium(I) carbonyl sawhorse complexes were prepared and structurally characterized, including the first sawhorse complex that contains two different carboxylate ligands and the first two sawhorse complexes containing only one tri-*p*-tolylphosphane ligand. A total of eight crystal structures of Os_2 sawhorse complexes have been reported herein, and the Os—Os bond distances in these complexes range from 2.7419 (3) to 2.7677 (3) Å. Several factors may influence the length of the Os—Os single bonds, but it appears as if torsional twisting around the Os—Os axis has a more significant effect than the basicity of the carboxylate ligands or the number of axial phosphane ligands. In general, as the average C_{eq}—Os—Os—C_{eq} torsion angle increases, the Os—Os bond distance decreases. More structural data involving additional sawhorse complexes is needed to confirm this trend, but it is noteworthy that the complex $Os_2(\mu$-pivalate$)_2(CO)_4(PPh_3)_2$ contains the largest average C_{eq}—Os—Os—C_{eq} torsion angle (25°) and the shortest Os—

Os bond length (2.7198 Å) of any Os_2 sawhorse complex to date (Chor *et al.*, 2016).

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References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.

Bruker (2008). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (2009). *SAINT*. Bruker AXS Inc, Madison, Wisconsin, USA.

Bullitt, J. G. & Cotton, F. A. (1971). *Inorg. Chim. Acta*, **5**, 406–412.

Chor, B. Y., Ganguly, R. & Leong, W. K. (2016). *J. Organomet. Chem.* **804**, 114–117.

Crooks, G. R., Johnson, B. F. G., Lewis, J., Williams, I. G. & Gamlen, G. (1969). *J. Chem. Soc. A*, pp. 2761–2766.

Deeming, A. J., Randle, N. P., Hursthouse, M. B. & Short, R. L. (1987). *J. Chem. Soc. Dalton Trans.* pp. 2473–2477.

Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.

Fikes, A. G., Gwini, N., Yoon, S. H., Nesterov, V. N. & Powell, G. L. (2014). *J. Organomet. Chem.* **772–773**, 188–191.

Gwini, N., Marolf, D. M., Yoon, S. H., Fikes, A. G., Dugan, A. C., Powell, G. L., Lynch, V. M., Nesterov, V. N. & McCandless, G. T. (2017). *J. Organomet. Chem.* **849–850**, 324–331.

Higashi, T. (2001). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.

Pyper, K. J., Jung, J. Y., Newton, B. S., Nesterov, V. N. & Powell, G. L. (2013). *J. Organomet. Chem.* **723**, 103–107.

Rigaku (2008). *CrystalClear*. Rigaku Inc., The Woodlands, Texas, USA.

Rigaku OD (2015). *CrysAlis PRO*. Rigaku Corporation, Tokyo, Japan.

Rigaku OD (2017). *CrysAlis PRO*. Rigaku Corporation, Tokyo, Japan.

Sheldrick, G. M. (1990). *XP* in *SHELXTL/PC*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.

Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

supporting information

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Asymmetric diosmium sawhorse complexes

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Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2017) for 1@CH2Cl2, 4@1.5C8H10; *APEX2* (Bruker, 2009) for (2), (8); *CrystalClear* (Rigaku, 2008) for (3), 7@CH2Cl2; *CrysAlis PRO* (Rigaku OD, 2015) for (5), (6). Cell refinement: *CrysAlis PRO* (Rigaku OD, 2017) for 1@CH2Cl2, 4@1.5C8H10; *SAINT* (Bruker, 2009) for (2), (8); *CrystalClear* (Rigaku, 2008) for (3), 7@CH2Cl2; *CrysAlis PRO* (Rigaku OD, 2015) for (5), (6). Data reduction: *CrysAlis PRO* (Rigaku OD, 2017) for 1@CH2Cl2, 4@1.5C8H10; *SAINT* (Bruker, 2009) for (2), (8); *CrystalClear* (Rigaku, 2008) for (3), 7@CH2Cl2; *CrysAlis PRO* (Rigaku OD, 2015) for (5), (6). Program(s) used to solve structure: SHELXT (Sheldrick, 2015a) for 1@CH2Cl2, (2), (5), (6), (8); *SIR97* (Altomare *et al.*, 1999) for (3), 7@CH2Cl2; SHELXT2014 (Sheldrick, 2015a) for 4@1.5C8H10. Program(s) used to refine structure: *SHELXL2018* (Sheldrick, 2015b) for 1@CH2Cl2, (2), 4@1.5C8H10, (5), (6), (8); *SHELXL2013* (Sheldrick, 2015a) for (3); *SHELXL2013* (Sheldrick, 2013) for 7@CH2Cl2. Molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009) for 1@CH2Cl2, (2), 4@1.5C8H10, (5), (6), (8); *XP* in *SHELXTL/PC* (Sheldrick, 1990) for (3), 7@CH2Cl2. Software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009) for 1@CH2Cl2, (2), 4@1.5C8H10, (5), (6), (8); *XP* in *SHELXTL/PC* (Sheldrick, 1990) for (3), 7@CH2Cl2.

μ-Acetato-1*κO*:2*κO*′-*μ*-propanoato-1*κO*:2*κO*′-bis[tris(4-methylphenyl)phosphane]-1*κP*,2*κP*′-bis(dicarbonylosmium)(*Os*—*Os*) dichloromethane monosolvate (1@CH2Cl2)

Crystal data

$[Os_2(C_2H_3O_2)(C_3H_5O_2)(C_{21}H_{21}P)_2(CO)_4]\cdot CH_2Cl_2$
M_r = 1318.17
Monoclinic, $P2_1/c$
a = 18.5007 (4) Å
b = 18.5396 (4) Å
c = 14.8090 (3) Å
β = 91.9976 (17)°
V = 5076.35 (18) Å^3
Z = 4
$F(000)$ = 2576
D_x = 1.725 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 12326 reflections
θ = 4.5–73.3°
μ = 11.30 mm^{-1}
T = 100 K
Plank, colorless
0.22 × 0.06 × 0.03 mm

Data collection

Rigaku SuperNova AtlasS2 CCD diffractometer
Detector resolution: 5.2387 pixels mm^{-1}
ω scans
Absorption correction: gaussian (CrysAlis PRO; Rigaku OD, 2017)
T_{min} = 0.428, T_{max} = 1.000
20129 measured reflections
9265 independent reflections

8475 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 68.3°$, $\theta_{min} = 5.6°$

$h = -21 \rightarrow 22$
$k = -19 \rightarrow 22$
$l = -17 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.023$
$wR(F^2) = 0.055$
$S = 1.00$
9265 reflections
632 parameters
112 restraints

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0214P)^2 + 5.3085P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 1.16$ e Å^{-3}
$\Delta\rho_{min} = -1.05$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Os1	0.24602 (2)	0.48548 (2)	0.15721 (2)	0.01357 (4)	
Os2	0.23338 (2)	0.49256 (2)	0.34230 (2)	0.01296 (4)	
P1	0.25891 (4)	0.50855 (4)	−0.00045 (5)	0.01549 (15)	
P2	0.21444 (4)	0.53185 (4)	0.49394 (5)	0.01377 (14)	
O1	0.39063 (12)	0.41219 (15)	0.17787 (16)	0.0266 (5)	
O2	0.17142 (14)	0.34201 (14)	0.13616 (17)	0.0276 (5)	
O3	0.36509 (14)	0.40392 (17)	0.39324 (17)	0.0354 (6)	
O4	0.14255 (14)	0.35845 (14)	0.35453 (16)	0.0290 (5)	
O5	0.28772 (13)	0.59099 (13)	0.17664 (14)	0.0217 (5)	
O6	0.29035 (13)	0.59111 (14)	0.32777 (15)	0.0253 (5)	
O7	0.14672 (11)	0.54428 (13)	0.15336 (14)	0.0211 (5)	
O8	0.14444 (12)	0.55903 (14)	0.30353 (14)	0.0220 (5)	
C1	0.33496 (17)	0.44046 (18)	0.16955 (19)	0.0187 (6)	
C2	0.20033 (16)	0.39689 (19)	0.1439 (2)	0.0204 (7)	
C3	0.31432 (18)	0.4387 (2)	0.3731 (2)	0.0221 (7)	
C4	0.17734 (17)	0.40972 (19)	0.3493 (2)	0.0210 (7)	
C5	0.29965 (17)	0.62065 (17)	0.2519 (2)	0.0218 (6)	
C8	0.12202 (15)	0.57186 (17)	0.2239 (2)	0.0188 (6)	
C10	0.29294 (16)	0.56904 (16)	0.5577 (2)	0.0152 (6)	
C11	0.28798 (17)	0.58869 (19)	0.6477 (2)	0.0206 (7)	
H11	0.243680	0.581524	0.677009	0.025*	
C12	0.34607 (17)	0.6185 (2)	0.6957 (2)	0.0214 (7)	
H12	0.341304	0.631017	0.757372	0.026*	
C13	0.41184 (16)	0.63028 (18)	0.6541 (2)	0.0190 (6)	
C14	0.41764 (17)	0.6085 (2)	0.5653 (2)	0.0222 (7)	
H14	0.462308	0.614585	0.536482	0.027*	
C15	0.35921 (17)	0.57797 (19)	0.5174 (2)	0.0218 (7)	

H15	0.364641	0.563058	0.456666	0.026*
C16	0.47439 (17)	0.6672 (2)	0.7040 (2)	0.0244 (7)
H16A	0.470386	0.719545	0.695725	0.037*
H16B	0.520114	0.650346	0.680050	0.037*
H16C	0.473149	0.655727	0.768528	0.037*
C17	0.14668 (16)	0.60298 (17)	0.49880 (19)	0.0154 (6)
C18	0.07374 (17)	0.58515 (19)	0.4842 (2)	0.0206 (6)
H18	0.060220	0.536103	0.475853	0.025*
C19	0.02107 (17)	0.6382 (2)	0.4817 (2)	0.0227 (7)
H19	−0.028264	0.624888	0.472896	0.027*
C20	0.03913 (18)	0.7107 (2)	0.4918 (2)	0.0223 (7)
C21	0.11192 (19)	0.72843 (19)	0.5041 (2)	0.0235 (7)
H21	0.125531	0.777660	0.510092	0.028*
C22	0.16513 (17)	0.67526 (19)	0.5079 (2)	0.0204 (6)
H22	0.214458	0.688563	0.516747	0.024*
C23	−0.0188 (2)	0.7680 (2)	0.4890 (3)	0.0327 (8)
H23A	−0.049023	0.762871	0.541756	0.049*
H23B	−0.048956	0.762628	0.433694	0.049*
H23C	0.003800	0.815822	0.489889	0.049*
C24	0.18198 (15)	0.46346 (18)	0.5708 (2)	0.0157 (6)
C25	0.20280 (16)	0.39197 (18)	0.5619 (2)	0.0173 (6)
H25	0.231485	0.378490	0.512623	0.021*
C26	0.18271 (16)	0.33976 (18)	0.6231 (2)	0.0180 (6)
H26	0.196732	0.291015	0.614533	0.022*
C27	0.14207 (16)	0.35819 (19)	0.6974 (2)	0.0197 (6)
C28	0.12092 (17)	0.42973 (19)	0.7069 (2)	0.0200 (6)
H28	0.093335	0.443309	0.757079	0.024*
C29	0.13952 (16)	0.48160 (17)	0.6441 (2)	0.0175 (6)
H29	0.123336	0.529865	0.650930	0.021*
C30	0.1246 (2)	0.3029 (2)	0.7678 (2)	0.0283 (8)
H30A	0.088624	0.268937	0.742620	0.042*
H30B	0.105160	0.327067	0.820513	0.042*
H30C	0.168693	0.276563	0.786020	0.042*
C31	0.33106 (17)	0.57266 (19)	−0.0201 (2)	0.0186 (6)
C32	0.32189 (18)	0.6459 (2)	−0.0017 (2)	0.0253 (7)
H32	0.276132	0.662835	0.016272	0.030*
C33	0.3785 (2)	0.6941 (2)	−0.0092 (3)	0.0325 (8)
H33	0.370747	0.743849	0.002209	0.039*
C34	0.4469 (2)	0.6705 (2)	−0.0335 (2)	0.0291 (8)
C35	0.45630 (17)	0.5974 (2)	−0.0506 (2)	0.0254 (7)
H35	0.502391	0.580222	−0.067077	0.030*
C36	0.39947 (16)	0.54921 (19)	−0.0440 (2)	0.0199 (6)
H36	0.407204	0.499497	−0.055853	0.024*
C37	0.5092 (2)	0.7229 (3)	−0.0398 (3)	0.0418 (10)
H37A	0.524938	0.738388	0.021061	0.063*
H37B	0.493568	0.765050	−0.075331	0.063*
H37C	0.549566	0.699297	−0.069177	0.063*
C38	0.28317 (16)	0.43215 (18)	−0.0719 (2)	0.0182 (6)

C39	0.29060 (16)	0.44399 (19)	−0.1646 (2)	0.0198 (6)	
H39	0.281898	0.490645	−0.189198	0.024*	
C40	0.31044 (17)	0.3884 (2)	−0.2204 (2)	0.0228 (7)	
H40	0.315867	0.397591	−0.282905	0.027*	
C41	0.32270 (17)	0.3190 (2)	−0.1872 (2)	0.0243 (7)	
C42	0.31542 (19)	0.3075 (2)	−0.0951 (2)	0.0268 (7)	
H42	0.323761	0.260721	−0.070694	0.032*	
C43	0.29603 (18)	0.3638 (2)	−0.0380 (2)	0.0239 (7)	
H43	0.291638	0.354882	0.024786	0.029*	
C44	0.3426 (2)	0.2582 (2)	−0.2490 (3)	0.0353 (9)	
H44A	0.298789	0.239353	−0.279777	0.053*	
H44B	0.366032	0.219575	−0.213472	0.053*	
H44C	0.375911	0.275952	−0.293940	0.053*	
C45	0.17989 (16)	0.54470 (18)	−0.0635 (2)	0.0176 (6)	
C46	0.18411 (17)	0.59718 (19)	−0.1303 (2)	0.0213 (7)	
H46	0.229091	0.619892	−0.140735	0.026*	
C47	0.12309 (19)	0.6165 (2)	−0.1818 (2)	0.0247 (7)	
H47	0.126739	0.652924	−0.226577	0.030*	
C48	0.05649 (18)	0.5835 (2)	−0.1689 (2)	0.0251 (7)	
C49	0.05235 (17)	0.5325 (2)	−0.1011 (2)	0.0234 (7)	
H49	0.007187	0.510349	−0.090312	0.028*	
C50	0.11293 (17)	0.51307 (19)	−0.0485 (2)	0.0194 (6)	
H50	0.108708	0.478064	−0.002159	0.023*	
C51	−0.0085 (2)	0.6020 (2)	−0.2291 (3)	0.0339 (9)	
H51A	−0.044745	0.563639	−0.225184	0.051*	
H51B	0.006362	0.606502	−0.291712	0.051*	
H51C	−0.029325	0.647717	−0.209418	0.051*	
C6	0.3157 (7)	0.7001 (3)	0.2532 (14)	0.0286 (17)	0.5
H6A	0.342512	0.714118	0.199177	0.034*	0.5
H6B	0.345090	0.713084	0.307904	0.034*	0.5
C6A	0.0584 (7)	0.6216 (7)	0.211 (2)	0.0189 (16)	0.5
H6AA	0.018924	0.607821	0.250609	0.023*	0.5
H6AB	0.039896	0.620471	0.147180	0.023*	0.5
C7	0.2419 (6)	0.7378 (6)	0.2532 (8)	0.063 (2)	0.5
H7A	0.214154	0.725351	0.197841	0.095*	0.5
H7B	0.249011	0.790100	0.256045	0.095*	0.5
H7C	0.215462	0.721881	0.305863	0.095*	0.5
C7A	0.0886 (4)	0.6989 (4)	0.2361 (5)	0.0257 (13)	0.5
H7AA	0.106583	0.698975	0.299141	0.039*	0.5
H7AB	0.049730	0.734591	0.228465	0.039*	0.5
H7AC	0.128106	0.711250	0.196543	0.039*	0.5
C9	0.0655 (7)	0.6299 (7)	0.214 (2)	0.021 (2)	0.5
H9A	0.089199	0.676991	0.210375	0.031*	0.5
H9B	0.034882	0.629016	0.266930	0.031*	0.5
H9C	0.035730	0.621372	0.159332	0.031*	0.5
C9A	0.3324 (6)	0.6948 (3)	0.2543 (14)	0.033 (2)	0.5
H9AA	0.333486	0.713028	0.316480	0.050*	0.5
H9AB	0.303331	0.727181	0.215434	0.050*	0.5

H9AC	0.381832	0.692543	0.232696	0.050*	0.5
Cl1	0.48305 (6)	0.58572 (8)	0.18071 (9)	0.0549 (3)	
Cl2	0.53460 (8)	0.58296 (8)	0.37000 (8)	0.0608 (3)	
C1S	0.5169 (2)	0.5330 (2)	0.2713 (3)	0.0395 (9)	
H1SA	0.481463	0.494723	0.284202	0.047*	
H1SB	0.562162	0.509281	0.253386	0.047*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Os1	0.01404 (7)	0.01543 (8)	0.01151 (7)	0.00011 (5)	0.00434 (4)	−0.00057 (5)
Os2	0.01369 (7)	0.01444 (7)	0.01095 (7)	0.00000 (5)	0.00317 (4)	−0.00072 (4)
P1	0.0169 (3)	0.0175 (4)	0.0124 (3)	0.0012 (3)	0.0049 (3)	0.0001 (3)
P2	0.0151 (3)	0.0143 (4)	0.0121 (3)	−0.0008 (3)	0.0034 (2)	−0.0010 (3)
O1	0.0203 (11)	0.0335 (14)	0.0263 (12)	0.0080 (10)	0.0039 (9)	0.0033 (10)
O2	0.0334 (13)	0.0205 (13)	0.0295 (13)	−0.0090 (11)	0.0077 (10)	−0.0056 (10)
O3	0.0286 (13)	0.0515 (18)	0.0260 (13)	0.0224 (13)	−0.0002 (10)	−0.0031 (12)
O4	0.0401 (14)	0.0239 (14)	0.0227 (12)	−0.0141 (11)	−0.0018 (10)	0.0025 (10)
O5	0.0304 (11)	0.0176 (11)	0.0176 (10)	−0.0033 (9)	0.0062 (9)	0.0000 (8)
O6	0.0334 (12)	0.0223 (12)	0.0204 (11)	−0.0097 (10)	0.0047 (9)	−0.0003 (9)
O7	0.0204 (10)	0.0292 (13)	0.0140 (10)	0.0079 (9)	0.0044 (8)	0.0002 (9)
O8	0.0236 (10)	0.0280 (13)	0.0144 (10)	0.0109 (9)	0.0043 (8)	0.0005 (9)
C1	0.0218 (15)	0.0235 (17)	0.0112 (14)	−0.0058 (13)	0.0052 (11)	−0.0006 (12)
C2	0.0169 (14)	0.0287 (19)	0.0157 (15)	0.0035 (13)	0.0049 (11)	−0.0005 (13)
C3	0.0264 (16)	0.0301 (19)	0.0102 (14)	−0.0017 (14)	0.0037 (12)	−0.0056 (13)
C4	0.0207 (15)	0.0274 (19)	0.0150 (15)	0.0039 (14)	0.0015 (11)	0.0001 (13)
C5	0.0217 (14)	0.0228 (15)	0.0213 (13)	−0.0045 (12)	0.0070 (11)	0.0020 (12)
C8	0.0170 (12)	0.0218 (14)	0.0178 (13)	0.0010 (11)	0.0031 (10)	0.0017 (11)
C10	0.0164 (13)	0.0123 (14)	0.0170 (14)	0.0011 (11)	0.0009 (11)	0.0013 (11)
C11	0.0176 (14)	0.0292 (18)	0.0155 (15)	−0.0015 (13)	0.0068 (11)	−0.0009 (13)
C12	0.0216 (15)	0.0292 (19)	0.0137 (14)	−0.0021 (13)	0.0041 (12)	−0.0009 (13)
C13	0.0176 (14)	0.0178 (16)	0.0214 (15)	0.0003 (12)	0.0000 (12)	0.0003 (12)
C14	0.0167 (14)	0.0308 (19)	0.0194 (15)	−0.0034 (13)	0.0043 (12)	−0.0019 (13)
C15	0.0221 (15)	0.0274 (18)	0.0163 (15)	−0.0023 (13)	0.0058 (12)	−0.0045 (13)
C16	0.0200 (15)	0.0319 (19)	0.0210 (16)	−0.0016 (14)	−0.0011 (12)	−0.0046 (14)
C17	0.0176 (14)	0.0177 (16)	0.0112 (13)	0.0013 (12)	0.0044 (11)	0.0005 (11)
C18	0.0209 (15)	0.0207 (17)	0.0204 (15)	−0.0020 (13)	0.0038 (12)	−0.0024 (13)
C19	0.0161 (14)	0.0316 (19)	0.0206 (15)	0.0049 (13)	0.0006 (11)	−0.0021 (14)
C20	0.0275 (17)	0.0259 (18)	0.0138 (15)	0.0086 (14)	0.0038 (12)	−0.0020 (13)
C21	0.0300 (17)	0.0179 (17)	0.0225 (16)	0.0029 (14)	0.0024 (13)	0.0003 (13)
C22	0.0192 (14)	0.0211 (17)	0.0211 (16)	−0.0012 (13)	0.0034 (12)	0.0003 (13)
C23	0.0331 (18)	0.031 (2)	0.034 (2)	0.0141 (16)	0.0027 (15)	−0.0015 (16)
C24	0.0144 (13)	0.0179 (16)	0.0149 (14)	−0.0024 (12)	0.0001 (10)	0.0004 (12)
C25	0.0166 (14)	0.0203 (16)	0.0150 (14)	−0.0005 (12)	0.0017 (11)	−0.0012 (12)
C26	0.0195 (14)	0.0160 (15)	0.0185 (15)	0.0022 (12)	0.0020 (11)	0.0016 (12)
C27	0.0174 (14)	0.0246 (17)	0.0173 (15)	−0.0021 (13)	0.0026 (11)	0.0032 (13)
C28	0.0215 (14)	0.0229 (17)	0.0163 (14)	−0.0039 (13)	0.0084 (11)	0.0001 (12)
C29	0.0211 (14)	0.0161 (15)	0.0155 (14)	0.0007 (12)	0.0032 (11)	−0.0025 (12)

C30	0.0313 (18)	0.0267 (19)	0.0276 (18)	0.0020 (15)	0.0097 (14)	0.0102 (15)
C31	0.0192 (14)	0.0238 (17)	0.0130 (14)	−0.0009 (13)	0.0046 (11)	0.0004 (12)
C32	0.0227 (16)	0.0243 (18)	0.0298 (18)	−0.0013 (14)	0.0120 (13)	−0.0014 (14)
C33	0.036 (2)	0.027 (2)	0.035 (2)	−0.0071 (16)	0.0158 (16)	−0.0067 (16)
C34	0.0285 (17)	0.036 (2)	0.0235 (17)	−0.0094 (16)	0.0093 (14)	−0.0027 (15)
C35	0.0181 (15)	0.039 (2)	0.0198 (16)	−0.0037 (14)	0.0052 (12)	0.0003 (14)
C36	0.0191 (14)	0.0253 (17)	0.0154 (14)	0.0031 (13)	0.0039 (11)	−0.0001 (12)
C37	0.035 (2)	0.045 (3)	0.046 (2)	−0.0175 (19)	0.0157 (18)	−0.008 (2)
C38	0.0144 (13)	0.0227 (17)	0.0175 (15)	0.0011 (12)	0.0037 (11)	−0.0046 (12)
C39	0.0176 (14)	0.0239 (17)	0.0180 (15)	0.0022 (12)	0.0017 (11)	0.0000 (13)
C40	0.0194 (15)	0.033 (2)	0.0158 (15)	0.0035 (14)	0.0012 (12)	−0.0054 (13)
C41	0.0199 (15)	0.0294 (19)	0.0233 (17)	0.0059 (14)	−0.0012 (12)	−0.0074 (14)
C42	0.0335 (18)	0.0208 (18)	0.0261 (18)	0.0065 (14)	0.0022 (14)	−0.0035 (14)
C43	0.0259 (16)	0.0259 (18)	0.0202 (16)	0.0049 (14)	0.0046 (12)	0.0000 (14)
C44	0.040 (2)	0.039 (2)	0.0268 (19)	0.0196 (18)	−0.0029 (15)	−0.0118 (16)
C45	0.0193 (14)	0.0196 (16)	0.0143 (14)	0.0038 (12)	0.0046 (11)	−0.0016 (12)
C46	0.0222 (15)	0.0220 (17)	0.0203 (15)	0.0034 (13)	0.0080 (12)	0.0009 (13)
C47	0.0291 (17)	0.0248 (18)	0.0206 (16)	0.0087 (14)	0.0062 (13)	0.0022 (13)
C48	0.0235 (16)	0.0309 (19)	0.0209 (16)	0.0086 (14)	0.0005 (13)	−0.0056 (14)
C49	0.0189 (14)	0.0318 (19)	0.0198 (15)	0.0004 (14)	0.0060 (12)	−0.0065 (14)
C50	0.0202 (14)	0.0267 (18)	0.0116 (14)	0.0024 (13)	0.0047 (11)	−0.0020 (12)
C51	0.0270 (17)	0.045 (2)	0.0293 (19)	0.0083 (17)	−0.0011 (14)	0.0026 (17)
C6	0.036 (4)	0.023 (3)	0.027 (3)	−0.012 (3)	0.005 (4)	0.001 (2)
C6A	0.018 (3)	0.021 (3)	0.018 (3)	0.003 (2)	0.001 (3)	−0.002 (3)
C7	0.071 (5)	0.042 (4)	0.077 (5)	0.008 (4)	0.019 (5)	−0.001 (4)
C7A	0.030 (3)	0.020 (3)	0.027 (3)	0.004 (2)	0.000 (3)	−0.003 (3)
C9	0.017 (3)	0.026 (4)	0.019 (4)	0.004 (3)	−0.001 (3)	−0.002 (4)
C9A	0.043 (5)	0.021 (3)	0.036 (4)	−0.008 (3)	0.020 (5)	−0.005 (3)
Cl1	0.0407 (5)	0.0619 (8)	0.0615 (7)	0.0047 (5)	−0.0063 (5)	0.0222 (6)
Cl2	0.0862 (9)	0.0585 (8)	0.0396 (6)	−0.0064 (7)	0.0283 (6)	−0.0143 (5)
C1S	0.045 (2)	0.030 (2)	0.044 (2)	−0.0006 (18)	0.0034 (18)	0.0017 (18)

Geometric parameters (Å, º)

Os1—Os2	2.7623 (2)	C30—H30A	0.9800
Os1—P1	2.3937 (7)	C30—H30B	0.9800
Os1—O5	2.119 (2)	C30—H30C	0.9800
Os1—O7	2.135 (2)	C31—C32	1.396 (5)
Os1—C1	1.848 (3)	C31—C36	1.395 (4)
Os1—C2	1.854 (4)	C32—H32	0.9500
Os2—P2	2.3981 (7)	C32—C33	1.384 (5)
Os2—O6	2.124 (2)	C33—H33	0.9500
Os2—O8	2.119 (2)	C33—C34	1.398 (5)
Os2—C3	1.844 (3)	C34—C35	1.390 (6)
Os2—C4	1.858 (4)	C34—C37	1.514 (5)
P1—C31	1.818 (3)	C35—H35	0.9500
P1—C38	1.833 (3)	C35—C36	1.386 (5)
P1—C45	1.834 (3)	C36—H36	0.9500

P2—C10	1.839 (3)	C37—H37A	0.9800
P2—C17	1.823 (3)	C37—H37B	0.9800
P2—C24	1.820 (3)	C37—H37C	0.9800
O1—C1	1.158 (4)	C38—C39	1.402 (4)
O2—C2	1.153 (4)	C38—C43	1.382 (5)
O3—C3	1.169 (4)	C39—H39	0.9500
O4—C4	1.152 (4)	C39—C40	1.379 (5)
O5—C5	1.255 (4)	C40—H40	0.9500
O6—C5	1.268 (4)	C40—C41	1.393 (5)
O7—C8	1.263 (4)	C41—C42	1.392 (5)
O8—C8	1.259 (4)	C41—C44	1.506 (5)
C5—C6	1.502 (4)	C42—H42	0.9500
C5—C9A	1.502 (4)	C42—C43	1.398 (5)
C8—C6A	1.502 (4)	C43—H43	0.9500
C8—C9	1.502 (4)	C44—H44A	0.9800
C10—C11	1.388 (4)	C44—H44B	0.9800
C10—C15	1.392 (4)	C44—H44C	0.9800
C11—H11	0.9500	C45—C46	1.392 (5)
C11—C12	1.382 (5)	C45—C50	1.395 (5)
C12—H12	0.9500	C46—H46	0.9500
C12—C13	1.400 (4)	C46—C47	1.387 (5)
C13—C14	1.383 (5)	C47—H47	0.9500
C13—C16	1.515 (4)	C47—C48	1.395 (5)
C14—H14	0.9500	C48—C49	1.384 (5)
C14—C15	1.393 (5)	C48—C51	1.510 (5)
C15—H15	0.9500	C49—H49	0.9500
C16—H16A	0.9800	C49—C50	1.390 (5)
C16—H16B	0.9800	C50—H50	0.9500
C16—H16C	0.9800	C51—H51A	0.9800
C17—C18	1.399 (4)	C51—H51B	0.9800
C17—C22	1.388 (5)	C51—H51C	0.9800
C18—H18	0.9500	C6—H6A	0.9900
C18—C19	1.384 (5)	C6—H6B	0.9900
C19—H19	0.9500	C6—C7	1.533 (17)
C19—C20	1.392 (5)	C6A—H6AA	0.9900
C20—C21	1.392 (5)	C6A—H6AB	0.9900
C20—C23	1.510 (5)	C6A—C7A	1.579 (18)
C21—H21	0.9500	C7—H7A	0.9800
C21—C22	1.393 (5)	C7—H7B	0.9800
C22—H22	0.9500	C7—H7C	0.9800
C23—H23A	0.9800	C7A—H7AA	0.9800
C23—H23B	0.9800	C7A—H7AB	0.9800
C23—H23C	0.9800	C7A—H7AC	0.9800
C24—C25	1.388 (5)	C9—H9A	0.9800
C24—C29	1.404 (4)	C9—H9B	0.9800
C25—H25	0.9500	C9—H9C	0.9800
C25—C26	1.386 (4)	C9A—H9AA	0.9800
C26—H26	0.9500	C9A—H9AB	0.9800

C26—C27	1.396 (4)	C9A—H9AC	0.9800
C27—C28	1.391 (5)	Cl1—C1S	1.758 (4)
C27—C30	1.505 (5)	Cl2—C1S	1.751 (4)
C28—H28	0.9500	C1S—H1SA	0.9900
C28—C29	1.389 (5)	C1S—H1SB	0.9900
C29—H29	0.9500		

P1—Os1—Os2	166.94 (2)	C24—C29—H29	119.7
O5—Os1—Os2	82.20 (6)	C28—C29—C24	120.7 (3)
O5—Os1—P1	85.37 (6)	C28—C29—H29	119.7
O5—Os1—O7	80.86 (9)	C27—C30—H30A	109.5
O7—Os1—Os2	84.24 (6)	C27—C30—H30B	109.5
O7—Os1—P1	89.86 (6)	C27—C30—H30C	109.5
C1—Os1—Os2	91.67 (9)	H30A—C30—H30B	109.5
C1—Os1—P1	93.36 (9)	H30A—C30—H30C	109.5
C1—Os1—O5	94.86 (12)	H30B—C30—H30C	109.5
C1—Os1—O7	174.43 (11)	C32—C31—P1	120.6 (2)
C1—Os1—C2	90.66 (14)	C32—C31—C36	118.0 (3)
C2—Os1—Os2	95.37 (9)	C36—C31—P1	121.0 (3)
C2—Os1—P1	96.60 (10)	C31—C32—H32	119.5
C2—Os1—O5	174.02 (11)	C33—C32—C31	121.0 (3)
C2—Os1—O7	93.48 (12)	C33—C32—H32	119.5
P2—Os2—Os1	164.55 (2)	C32—C33—H33	119.6
O6—Os2—Os1	83.16 (6)	C32—C33—C34	120.8 (4)
O6—Os2—P2	85.57 (6)	C34—C33—H33	119.6
O8—Os2—Os1	81.41 (6)	C33—C34—C37	120.9 (4)
O8—Os2—P2	86.54 (6)	C35—C34—C33	118.2 (3)
O8—Os2—O6	81.70 (10)	C35—C34—C37	121.0 (3)
C3—Os2—Os1	97.08 (9)	C34—C35—H35	119.5
C3—Os2—P2	94.43 (9)	C36—C35—C34	121.0 (3)
C3—Os2—O6	95.14 (13)	C36—C35—H35	119.5
C3—Os2—O8	176.62 (13)	C31—C36—H36	119.5
C3—Os2—C4	89.32 (15)	C35—C36—C31	120.9 (3)
C4—Os2—Os1	94.74 (9)	C35—C36—H36	119.5
C4—Os2—P2	95.67 (10)	C34—C37—H37A	109.5
C4—Os2—O6	175.27 (12)	C34—C37—H37B	109.5
C4—Os2—O8	93.80 (12)	C34—C37—H37C	109.5
C31—P1—Os1	111.82 (10)	H37A—C37—H37B	109.5
C31—P1—C38	102.51 (14)	H37A—C37—H37C	109.5
C31—P1—C45	104.85 (15)	H37B—C37—H37C	109.5
C38—P1—Os1	117.36 (11)	C39—C38—P1	118.6 (3)
C45—P1—Os1	117.19 (10)	C43—C38—P1	122.8 (2)
C45—P1—C38	101.28 (14)	C43—C38—C39	118.6 (3)
C10—P2—Os2	117.15 (10)	C38—C39—H39	119.8
C17—P2—Os2	112.33 (10)	C40—C39—C38	120.5 (3)
C17—P2—C10	103.91 (14)	C40—C39—H39	119.8
C24—P2—Os2	115.85 (10)	C39—C40—H40	119.2
C24—P2—C10	102.25 (13)	C39—C40—C41	121.5 (3)

C24—P2—C17	103.71 (14)	C41—C40—H40	119.2
C5—O5—Os1	125.2 (2)	C40—C41—C44	121.2 (3)
C5—O6—Os2	123.0 (2)	C42—C41—C40	117.9 (3)
C8—O7—Os1	121.36 (19)	C42—C41—C44	121.0 (3)
C8—O8—Os2	126.24 (19)	C41—C42—H42	119.5
O1—C1—Os1	179.6 (3)	C41—C42—C43	121.0 (3)
O2—C2—Os1	179.3 (3)	C43—C42—H42	119.5
O3—C3—Os2	179.1 (3)	C38—C43—C42	120.6 (3)
O4—C4—Os2	179.3 (3)	C38—C43—H43	119.7
O5—C5—O6	124.9 (3)	C42—C43—H43	119.7
O5—C5—C6	118.0 (8)	C41—C44—H44A	109.5
O5—C5—C9A	118.7 (9)	C41—C44—H44B	109.5
O6—C5—C6	116.4 (8)	C41—C44—H44C	109.5
O6—C5—C9A	116.2 (9)	H44A—C44—H44B	109.5
O7—C8—C6A	116.4 (12)	H44A—C44—H44C	109.5
O7—C8—C9	118.8 (12)	H44B—C44—H44C	109.5
O8—C8—O7	125.5 (3)	C46—C45—P1	123.6 (2)
O8—C8—C6A	118.0 (12)	C46—C45—C50	118.6 (3)
O8—C8—C9	115.4 (11)	C50—C45—P1	117.5 (2)
C11—C10—P2	120.9 (2)	C45—C46—H46	119.8
C11—C10—C15	118.0 (3)	C47—C46—C45	120.5 (3)
C15—C10—P2	121.1 (2)	C47—C46—H46	119.8
C10—C11—H11	119.3	C46—C47—H47	119.4
C12—C11—C10	121.5 (3)	C46—C47—C48	121.2 (3)
C12—C11—H11	119.3	C48—C47—H47	119.4
C11—C12—H12	119.7	C47—C48—C51	120.6 (3)
C11—C12—C13	120.6 (3)	C49—C48—C47	118.1 (3)
C13—C12—H12	119.7	C49—C48—C51	121.3 (3)
C12—C13—C16	121.2 (3)	C48—C49—H49	119.3
C14—C13—C12	118.0 (3)	C48—C49—C50	121.4 (3)
C14—C13—C16	120.8 (3)	C50—C49—H49	119.3
C13—C14—H14	119.4	C45—C50—H50	119.8
C13—C14—C15	121.2 (3)	C49—C50—C45	120.3 (3)
C15—C14—H14	119.4	C49—C50—H50	119.8
C10—C15—C14	120.7 (3)	C48—C51—H51A	109.5
C10—C15—H15	119.7	C48—C51—H51B	109.5
C14—C15—H15	119.7	C48—C51—H51C	109.5
C13—C16—H16A	109.5	H51A—C51—H51B	109.5
C13—C16—H16B	109.5	H51A—C51—H51C	109.5
C13—C16—H16C	109.5	H51B—C51—H51C	109.5
H16A—C16—H16B	109.5	C5—C6—H6A	110.6
H16A—C16—H16C	109.5	C5—C6—H6B	110.6
H16B—C16—H16C	109.5	C5—C6—C7	105.7 (8)
C18—C17—P2	118.9 (3)	H6A—C6—H6B	108.7
C22—C17—P2	122.3 (2)	C7—C6—H6A	110.6
C22—C17—C18	118.4 (3)	C7—C6—H6B	110.6
C17—C18—H18	119.6	C8—C6A—H6AA	110.8
C19—C18—C17	120.7 (3)	C8—C6A—H6AB	110.8

C19—C18—H18	119.6	C8—C6A—C7A	105.0 (9)
C18—C19—H19	119.4	H6AA—C6A—H6AB	108.8
C18—C19—C20	121.1 (3)	C7A—C6A—H6AA	110.8
C20—C19—H19	119.4	C7A—C6A—H6AB	110.8
C19—C20—C23	120.6 (3)	C6—C7—H7A	109.5
C21—C20—C19	118.0 (3)	C6—C7—H7B	109.5
C21—C20—C23	121.3 (3)	C6—C7—H7C	109.5
C20—C21—H21	119.4	H7A—C7—H7B	109.5
C20—C21—C22	121.1 (3)	H7A—C7—H7C	109.5
C22—C21—H21	119.4	H7B—C7—H7C	109.5
C17—C22—C21	120.5 (3)	C6A—C7A—H7AA	109.5
C17—C22—H22	119.7	C6A—C7A—H7AB	109.5
C21—C22—H22	119.7	C6A—C7A—H7AC	109.5
C20—C23—H23A	109.5	H7AA—C7A—H7AB	109.5
C20—C23—H23B	109.5	H7AA—C7A—H7AC	109.5
C20—C23—H23C	109.5	H7AB—C7A—H7AC	109.5
H23A—C23—H23B	109.5	C8—C9—H9A	109.5
H23A—C23—H23C	109.5	C8—C9—H9B	109.5
H23B—C23—H23C	109.5	C8—C9—H9C	109.5
C25—C24—P2	120.5 (2)	H9A—C9—H9B	109.5
C25—C24—C29	117.9 (3)	H9A—C9—H9C	109.5
C29—C24—P2	121.5 (2)	H9B—C9—H9C	109.5
C24—C25—H25	119.2	C5—C9A—H9AA	109.5
C26—C25—C24	121.5 (3)	C5—C9A—H9AB	109.5
C26—C25—H25	119.2	C5—C9A—H9AC	109.5
C25—C26—H26	119.7	H9AA—C9A—H9AB	109.5
C25—C26—C27	120.6 (3)	H9AA—C9A—H9AC	109.5
C27—C26—H26	119.7	H9AB—C9A—H9AC	109.5
C26—C27—C30	120.9 (3)	Cl1—C1S—H1SA	108.9
C28—C27—C26	118.3 (3)	Cl1—C1S—H1SB	108.9
C28—C27—C30	120.8 (3)	Cl2—C1S—Cl1	113.2 (3)
C27—C28—H28	119.5	Cl2—C1S—H1SA	108.9
C29—C28—C27	121.0 (3)	Cl2—C1S—H1SB	108.9
C29—C28—H28	119.5	H1SA—C1S—H1SB	107.7

Os1—P1—C31—C32	−73.8 (3)	C17—C18—C19—C20	1.3 (5)
Os1—P1—C31—C36	98.8 (2)	C18—C17—C22—C21	1.2 (5)
Os1—P1—C38—C39	179.6 (2)	C18—C19—C20—C21	0.3 (5)
Os1—P1—C38—C43	−1.9 (3)	C18—C19—C20—C23	−180.0 (3)
Os1—P1—C45—C46	141.0 (2)	C19—C20—C21—C22	−1.2 (5)
Os1—P1—C45—C50	−44.7 (3)	C20—C21—C22—C17	0.4 (5)
Os1—O5—C5—O6	2.0 (5)	C22—C17—C18—C19	−2.0 (5)
Os1—O5—C5—C6	−168.6 (6)	C23—C20—C21—C22	179.1 (3)
Os1—O5—C5—C9A	177.4 (6)	C24—P2—C10—C11	−48.0 (3)
Os1—O7—C8—O8	−12.2 (5)	C24—P2—C10—C15	131.6 (3)
Os1—O7—C8—C6A	170.6 (8)	C24—P2—C17—C18	−52.3 (3)
Os1—O7—C8—C9	162.1 (8)	C24—P2—C17—C22	134.1 (3)
Os2—P2—C10—C11	−175.8 (2)	C24—C25—C26—C27	−1.6 (5)

Os2—P2—C10—C15	3.9 (3)	C25—C24—C29—C28	1.8 (4)
Os2—P2—C17—C18	73.6 (2)	C25—C26—C27—C28	1.5 (5)
Os2—P2—C17—C22	−100.1 (3)	C25—C26—C27—C30	−175.6 (3)
Os2—P2—C24—C25	33.0 (3)	C26—C27—C28—C29	0.2 (5)
Os2—P2—C24—C29	−151.4 (2)	C27—C28—C29—C24	−1.8 (5)
Os2—O6—C5—O5	−12.3 (5)	C29—C24—C25—C26	−0.1 (4)
Os2—O6—C5—C6	158.4 (6)	C30—C27—C28—C29	177.3 (3)
Os2—O6—C5—C9A	172.2 (6)	C31—P1—C38—C39	−57.5 (3)
Os2—O8—C8—O7	3.7 (5)	C31—P1—C38—C43	121.0 (3)
Os2—O8—C8—C6A	−179.1 (8)	C31—P1—C45—C46	16.3 (3)
Os2—O8—C8—C9	−170.7 (8)	C31—P1—C45—C50	−169.4 (2)
P1—C31—C32—C33	174.5 (3)	C31—C32—C33—C34	−1.5 (6)
P1—C31—C36—C35	−173.8 (2)	C32—C31—C36—C35	−1.0 (5)
P1—C38—C39—C40	178.5 (2)	C32—C33—C34—C35	0.6 (6)
P1—C38—C43—C42	−179.0 (3)	C32—C33—C34—C37	−178.7 (4)
P1—C45—C46—C47	173.2 (3)	C33—C34—C35—C36	0.1 (5)
P1—C45—C50—C49	−173.0 (3)	C34—C35—C36—C31	0.1 (5)
P2—C10—C11—C12	−178.3 (3)	C36—C31—C32—C33	1.7 (5)
P2—C10—C15—C14	177.7 (3)	C37—C34—C35—C36	179.3 (3)
P2—C17—C18—C19	−175.9 (2)	C38—P1—C31—C32	159.6 (3)
P2—C17—C22—C21	174.9 (2)	C38—P1—C31—C36	−27.8 (3)
P2—C24—C25—C26	175.6 (2)	C38—P1—C45—C46	−90.0 (3)
P2—C24—C29—C28	−173.9 (2)	C38—P1—C45—C50	84.3 (3)
O5—C5—C6—C7	86.2 (12)	C38—C39—C40—C41	0.9 (5)
O6—C5—C6—C7	−85.2 (12)	C39—C38—C43—C42	−0.5 (5)
O7—C8—C6A—C7A	−111.4 (13)	C39—C40—C41—C42	−1.0 (5)
O8—C8—C6A—C7A	71.2 (17)	C39—C40—C41—C44	178.5 (3)
C10—P2—C17—C18	−158.9 (2)	C40—C41—C42—C43	0.4 (5)
C10—P2—C17—C22	27.5 (3)	C41—C42—C43—C38	0.4 (5)
C10—P2—C24—C25	−95.6 (3)	C43—C38—C39—C40	−0.1 (5)
C10—P2—C24—C29	80.0 (3)	C44—C41—C42—C43	−179.1 (3)
C10—C11—C12—C13	0.6 (5)	C45—P1—C31—C32	54.2 (3)
C11—C10—C15—C14	−2.6 (5)	C45—P1—C31—C36	−133.2 (3)
C11—C12—C13—C14	−2.7 (5)	C45—P1—C38—C39	50.7 (3)
C11—C12—C13—C16	176.0 (3)	C45—P1—C38—C43	−130.8 (3)
C12—C13—C14—C15	2.1 (5)	C45—C46—C47—C48	−0.9 (5)
C13—C14—C15—C10	0.6 (6)	C46—C45—C50—C49	1.6 (5)
C15—C10—C11—C12	2.1 (5)	C46—C47—C48—C49	2.2 (5)
C16—C13—C14—C15	−176.6 (3)	C46—C47—C48—C51	−176.4 (3)
C17—P2—C10—C11	59.7 (3)	C47—C48—C49—C50	−1.6 (5)
C17—P2—C10—C15	−120.7 (3)	C48—C49—C50—C45	−0.3 (5)
C17—P2—C24—C25	156.6 (2)	C50—C45—C46—C47	−1.1 (5)
C17—P2—C24—C29	−27.9 (3)	C51—C48—C49—C50	176.9 (3)

Bis(*μ*-acetato-1*κO*:2*κO′*)bis(tricarbonylosmium)(*Os—Os*) (2)

Crystal data

$[Os_2(C_2H_3O_2)_2(CO)_6]$
$M_r = 666.55$
Monoclinic, $P2_1/n$
$a = 7.6949$ (5) Å
$b = 14.3612$ (10) Å
$c = 13.8623$ (9) Å
$\beta = 105.202$ (1)°
$V = 1478.29$ (17) Å^3
$Z = 4$
$F(000) = 1192$
$D_x = 2.995$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9606 reflections
$\theta = 2.8$–$33.1°$
$\mu = 17.22$ mm^{-1}
$T = 200$ K
Block, colorless
0.19 × 0.12 × 0.07 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2008)
$T_{min} = 0.295$, $T_{max} = 0.747$
14002 measured reflections
3252 independent reflections
3003 reflections with $I > 2\sigma(I)$
$R_{int} = 0.046$
$\theta_{max} = 27.1°$, $\theta_{min} = 2.1°$
$h = -9 \rightarrow 9$
$k = -18 \rightarrow 18$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.023$
$wR(F^2) = 0.055$
$S = 1.09$
3252 reflections
202 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0082P)^2 + 2.0911P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.80$ e Å^{-3}
$\Delta\rho_{min} = -1.33$ e Å^{-3}
Extinction correction: SHELXL2018 (Sheldrick, 2015b), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00198 (9)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Os1	0.42547 (2)	0.22664 (2)	0.05236 (2)	0.01655 (7)
Os2	0.77231 (2)	0.21800 (2)	0.17010 (2)	0.01619 (7)
O8	0.6745 (4)	0.3123 (2)	0.2608 (2)	0.0223 (7)
O9	0.5294 (4)	0.3337 (2)	−0.0211 (2)	0.0239 (7)
O7	0.4059 (4)	0.3352 (2)	0.1523 (2)	0.0233 (7)
O10	0.8002 (4)	0.3386 (2)	0.0881 (2)	0.0253 (7)
O3	0.0418 (5)	0.2846 (3)	−0.0685 (3)	0.0424 (10)
O2	0.4741 (6)	0.0738 (3)	−0.0875 (3)	0.0430 (10)
O4	0.8856 (6)	0.0922 (3)	0.0236 (3)	0.0391 (9)

C7	0.5235 (6)	0.3522 (3)	0.2335 (3)	0.0190 (9)
O5	0.7127 (6)	0.0534 (3)	0.2927 (3)	0.0417 (9)
O1	0.3004 (6)	0.0859 (3)	0.1800 (3)	0.0462 (10)
O6	1.1610 (5)	0.2535 (4)	0.2995 (3)	0.0459 (10)
C4	0.8448 (6)	0.1390 (3)	0.0800 (3)	0.0262 (10)
C1	0.3453 (6)	0.1378 (3)	0.1300 (4)	0.0271 (10)
C5	0.7343 (6)	0.1147 (3)	0.2464 (3)	0.0243 (10)
C8	0.4787 (7)	0.4249 (3)	0.2994 (3)	0.0288 (10)
H8A	0.579579	0.432174	0.358999	0.043*
H8B	0.370674	0.406277	0.319369	0.043*
H8C	0.456292	0.484157	0.263170	0.043*
C9	0.6840 (6)	0.3691 (3)	0.0140 (3)	0.0211 (9)
C2	0.4554 (7)	0.1320 (3)	−0.0357 (3)	0.0269 (10)
C3	0.1837 (7)	0.2639 (3)	−0.0252 (4)	0.0260 (10)
C6	1.0190 (7)	0.2415 (4)	0.2537 (4)	0.0263 (10)
C10	0.7308 (8)	0.4540 (4)	−0.0362 (4)	0.0350 (12)
H10A	0.830534	0.486855	0.009782	0.053*
H10B	0.625795	0.495203	−0.055015	0.053*
H10C	0.766953	0.435746	−0.096241	0.053*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Os1	0.01386 (11)	0.01834 (11)	0.01733 (10)	0.00051 (6)	0.00388 (7)	−0.00074 (5)
Os2	0.01358 (11)	0.01921 (11)	0.01643 (10)	0.00390 (6)	0.00506 (7)	0.00252 (5)
O8	0.0225 (17)	0.0263 (16)	0.0179 (14)	0.0063 (13)	0.0052 (12)	−0.0054 (12)
O9	0.0249 (17)	0.0267 (17)	0.0201 (14)	−0.0001 (14)	0.0058 (13)	0.0088 (12)
O7	0.0233 (17)	0.0236 (16)	0.0233 (14)	0.0049 (13)	0.0064 (13)	−0.0021 (12)
O10	0.0228 (17)	0.0235 (17)	0.0281 (16)	−0.0032 (13)	0.0042 (13)	0.0067 (13)
O3	0.020 (2)	0.059 (3)	0.043 (2)	0.0106 (17)	−0.0002 (18)	−0.0021 (17)
O2	0.051 (2)	0.038 (2)	0.0347 (19)	0.0136 (19)	0.0013 (17)	−0.0148 (16)
O4	0.049 (2)	0.043 (2)	0.0332 (18)	0.0186 (19)	0.0241 (17)	−0.0037 (16)
C7	0.021 (2)	0.019 (2)	0.0194 (19)	−0.0005 (17)	0.0094 (17)	0.0012 (15)
O5	0.056 (3)	0.036 (2)	0.0348 (19)	−0.0006 (19)	0.0159 (18)	0.0141 (16)
O1	0.044 (2)	0.041 (2)	0.061 (3)	−0.0100 (19)	0.026 (2)	0.0172 (19)
O6	0.017 (2)	0.072 (3)	0.043 (2)	−0.001 (2)	−0.0022 (19)	0.007 (2)
C4	0.021 (2)	0.032 (3)	0.027 (2)	0.008 (2)	0.0091 (19)	0.0094 (19)
C1	0.017 (2)	0.029 (3)	0.034 (2)	−0.0016 (19)	0.005 (2)	−0.0031 (19)
C5	0.024 (2)	0.027 (2)	0.022 (2)	0.005 (2)	0.0078 (18)	0.0005 (18)
C8	0.036 (3)	0.026 (2)	0.027 (2)	0.000 (2)	0.014 (2)	−0.0064 (18)
C9	0.023 (2)	0.019 (2)	0.024 (2)	0.0017 (18)	0.0106 (18)	0.0015 (16)
C2	0.025 (2)	0.029 (2)	0.024 (2)	0.003 (2)	0.0008 (19)	−0.0003 (18)
C3	0.023 (3)	0.026 (2)	0.030 (2)	−0.0003 (19)	0.009 (2)	−0.0031 (18)
C6	0.025 (3)	0.031 (2)	0.025 (2)	0.004 (2)	0.012 (2)	0.0021 (19)
C10	0.040 (3)	0.030 (3)	0.038 (3)	−0.002 (2)	0.014 (2)	0.010 (2)

Geometric parameters (Å, º)

Os1—Os2	2.7419 (3)	O3—C3	1.139 (6)
Os1—O9	2.113 (3)	O2—C2	1.135 (6)
Os1—O7	2.118 (3)	O4—C4	1.137 (6)
Os1—C1	1.876 (5)	C7—C8	1.486 (6)
Os1—C2	1.881 (5)	O5—C5	1.128 (6)
Os1—C3	1.963 (5)	O1—C1	1.131 (6)
Os2—O8	2.116 (3)	O6—C6	1.124 (7)
Os2—O10	2.114 (3)	C8—H8A	0.9800
Os2—C4	1.877 (5)	C8—H8B	0.9800
Os2—C5	1.889 (5)	C8—H8C	0.9800
Os2—C6	1.976 (5)	C9—C10	1.494 (6)
O8—C7	1.262 (5)	C10—H10A	0.9800
O9—C9	1.268 (5)	C10—H10B	0.9800
O7—C7	1.269 (5)	C10—H10C	0.9800
O10—C9	1.251 (5)		
O9—Os1—Os2	83.54 (8)	C6—Os2—O10	87.60 (17)
O9—Os1—O7	83.14 (13)	C7—O8—Os2	123.7 (3)
O7—Os1—Os2	82.46 (8)	C9—O9—Os1	122.9 (3)
C1—Os1—Os2	92.46 (14)	C7—O7—Os1	124.4 (3)
C1—Os1—O9	174.06 (16)	C9—O10—Os2	125.3 (3)
C1—Os1—O7	92.00 (17)	O8—C7—O7	124.4 (4)
C1—Os1—C2	89.8 (2)	O8—C7—C8	118.6 (4)
C1—Os1—C3	95.3 (2)	O7—C7—C8	117.0 (4)
C2—Os1—Os2	95.05 (14)	O4—C4—Os2	178.3 (4)
C2—Os1—O9	94.95 (18)	O1—C1—Os1	177.4 (4)
C2—Os1—O7	177.01 (18)	O5—C5—Os2	179.3 (5)
C2—Os1—C3	95.8 (2)	C7—C8—H8A	109.5
C3—Os1—Os2	166.67 (14)	C7—C8—H8B	109.5
C3—Os1—O9	87.81 (17)	C7—C8—H8C	109.5
C3—Os1—O7	86.47 (17)	H8A—C8—H8B	109.5
O8—Os2—Os1	83.26 (8)	H8A—C8—H8C	109.5
O10—Os2—Os1	82.12 (8)	H8B—C8—H8C	109.5
O10—Os2—O8	84.12 (13)	O9—C9—C10	117.8 (4)
C4—Os2—Os1	92.53 (14)	O10—C9—O9	124.8 (4)
C4—Os2—O8	175.04 (16)	O10—C9—C10	117.4 (4)
C4—Os2—O10	92.72 (16)	O2—C2—Os1	178.7 (4)
C4—Os2—C5	90.8 (2)	O3—C3—Os1	178.3 (5)
C4—Os2—C6	94.9 (2)	O6—C6—Os2	178.2 (5)
C5—Os2—Os1	95.71 (14)	C9—C10—H10A	109.5
C5—Os2—O8	92.21 (16)	C9—C10—H10B	109.5
C5—Os2—O10	175.92 (16)	C9—C10—H10C	109.5
C5—Os2—C6	94.1 (2)	H10A—C10—H10B	109.5
C6—Os2—Os1	167.58 (15)	H10A—C10—H10C	109.5
C6—Os2—O8	88.79 (17)	H10B—C10—H10C	109.5

Bis(μ-acetato-1κO:2κO′)pentacarbonyl-1κ²C,2κ³C-[tris(4-methylphenyl)phosphane-1κP]diosmium(*Os*—*Os*) (3)

Crystal data

$[Os_2(C_2H_3O_2)_2(C_{21}H_{21}P)(CO)_5]$
M_r = 942.88
Triclinic, $P\overline{1}$
a = 10.452 (4) Å
b = 11.229 (4) Å
c = 13.045 (5) Å
α = 82.813 (6)°
β = 89.157 (8)°
γ = 89.457 (9)°
V = 1518.8 (10) Å^3
Z = 2
$F(000)$ = 892
D_x = 2.062 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4436 reflections
θ = 1.8–27.5°
μ = 8.46 mm^{-1}
T = 100 K
Prism, colorless
0.27 × 0.14 × 0.12 mm

Data collection

Rigaku SCX-Mini Mercury 2+ CCD diffractometer
Radiation source: sealed tube
ω–scans
Absorption correction: multi-scan (ABSCOR; Higashi, 2001)
T_{min} = 0.605, T_{max} = 1.00
32350 measured reflections
6915 independent reflections
6534 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 27.4°, θ_{min} = 3.0°
h = −13→13
k = −14→14
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.045
S = 1.02
6915 reflections
385 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.013P)^2 + 4.878P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 2.80 e Å^{-3}
$\Delta\rho_{min}$ = −1.05 e Å^{-3}
Extinction correction: SHELXL2013 (Sheldrick, 2015a), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00017 (6)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1347 (3)	0.4868 (3)	0.1894 (2)	0.0127 (6)
C2	0.0634 (3)	0.4462 (3)	0.2789 (2)	0.0145 (6)
H2	0.0367	0.5015	0.3245	0.017*
C3	0.0318 (3)	0.3263 (3)	0.3013 (2)	0.0157 (6)
H3	−0.0176	0.3007	0.3616	0.019*
C4	0.0714 (3)	0.2420 (3)	0.2368 (3)	0.0151 (6)
C5	0.1440 (3)	0.2823 (3)	0.1486 (3)	0.0168 (6)
H5	0.1730	0.2264	0.1042	0.020*

C6	0.1744 (3)	0.4033 (3)	0.1250 (2)	0.0143 (6)
H6	0.2229	0.4290	0.0642	0.017*
C7	0.0330 (4)	0.1124 (3)	0.2607 (3)	0.0238 (8)
H7A	0.0324	0.0893	0.3356	0.036*
H7B	0.0944	0.0618	0.2281	0.036*
H7C	−0.0527	0.1019	0.2338	0.036*
C8	0.2576 (3)	0.6603 (3)	0.0404 (2)	0.0125 (6)
C9	0.3909 (3)	0.6544 (4)	0.0417 (3)	0.0263 (8)
H9	0.4338	0.6500	0.1058	0.032*
C10	0.4618 (3)	0.6549 (4)	−0.0489 (3)	0.0275 (8)
H10	0.5525	0.6521	−0.0456	0.033*
C11	0.4039 (3)	0.6592 (3)	−0.1441 (2)	0.0159 (6)
C12	0.2701 (3)	0.6658 (3)	−0.1455 (3)	0.0209 (7)
H12	0.2274	0.6704	−0.2097	0.025*
C13	0.1983 (3)	0.6658 (3)	−0.0549 (3)	0.0198 (7)
H13	0.1076	0.6697	−0.0581	0.024*
C14	0.4829 (3)	0.6576 (3)	−0.2416 (3)	0.0215 (7)
H14A	0.5249	0.7353	−0.2590	0.032*
H14B	0.4273	0.6424	−0.2983	0.032*
H14C	0.5480	0.5940	−0.2308	0.032*
C15	0.0115 (3)	0.7157 (3)	0.1292 (2)	0.0123 (6)
C16	0.0042 (3)	0.8404 (3)	0.1045 (3)	0.0173 (7)
H16	0.0806	0.8861	0.0968	0.021*
C17	−0.1134 (3)	0.8983 (3)	0.0909 (3)	0.0186 (7)
H17	−0.1160	0.9830	0.0735	0.022*
C18	−0.2277 (3)	0.8350 (3)	0.1024 (2)	0.0180 (7)
C19	−0.2203 (3)	0.7104 (3)	0.1246 (3)	0.0182 (7)
H19	−0.2969	0.6650	0.1312	0.022*
C20	−0.1024 (3)	0.6512 (3)	0.1374 (3)	0.0168 (7)
H20	−0.0997	0.5661	0.1518	0.020*
C21	−0.3553 (3)	0.8995 (4)	0.0937 (3)	0.0271 (8)
H21A	−0.4068	0.8674	0.0414	0.041*
H21B	−0.3415	0.9855	0.0734	0.041*
H21C	−0.4003	0.8874	0.1606	0.041*
C22	0.4281 (3)	0.6810 (3)	0.2888 (2)	0.0152 (6)
C23	0.3029 (3)	0.8760 (3)	0.2091 (3)	0.0162 (6)
C24	0.2352 (3)	0.5818 (3)	0.5051 (3)	0.0148 (6)
C25	0.2099 (3)	0.4613 (3)	0.5673 (3)	0.0219 (7)
H25A	0.1252	0.4330	0.5505	0.033*
H25B	0.2127	0.4693	0.6412	0.033*
H25C	0.2753	0.4034	0.5505	0.033*
C26	0.0355 (3)	0.8338 (3)	0.4138 (2)	0.0113 (6)
C27	−0.1052 (3)	0.8548 (3)	0.4291 (3)	0.0161 (6)
H27A	−0.1508	0.8404	0.3667	0.024*
H27B	−0.1198	0.9379	0.4423	0.024*
H27C	−0.1368	0.7999	0.4881	0.024*
C28	0.3296 (3)	0.9928 (3)	0.4006 (3)	0.0169 (7)
C29	0.4871 (3)	0.8127 (3)	0.4656 (3)	0.0198 (7)

C30	0.3235 (3)	0.9004 (3)	0.6145 (3)	0.0176 (7)
O1	0.5308 (2)	0.6419 (2)	0.28700 (19)	0.0244 (6)
O2	0.3279 (2)	0.9606 (2)	0.1523 (2)	0.0252 (6)
O3	0.2162 (2)	0.5898 (2)	0.40969 (17)	0.0177 (5)
O4	0.2747 (2)	0.6648 (2)	0.55293 (18)	0.0173 (5)
O5	0.0720 (2)	0.7938 (2)	0.33261 (17)	0.0159 (5)
O6	0.1085 (2)	0.8585 (2)	0.48501 (17)	0.0141 (4)
O7	0.3398 (3)	1.0848 (2)	0.3535 (2)	0.0280 (6)
O8	0.5950 (2)	0.7955 (3)	0.4569 (2)	0.0335 (7)
O9	0.3396 (3)	0.9413 (2)	0.6878 (2)	0.0300 (6)
Os1	0.26266 (2)	0.74048 (2)	0.30025 (2)	0.01069 (4)
Os2	0.31080 (2)	0.84058 (2)	0.47863 (2)	0.01191 (4)
P1	0.16761 (7)	0.64666 (7)	0.16222 (6)	0.01095 (15)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0122 (14)	0.0121 (15)	0.0139 (15)	0.0001 (11)	−0.0031 (12)	−0.0014 (12)
C2	0.0146 (15)	0.0164 (16)	0.0136 (15)	−0.0008 (12)	0.0003 (12)	−0.0056 (12)
C3	0.0140 (15)	0.0195 (16)	0.0132 (15)	−0.0031 (12)	−0.0002 (12)	−0.0005 (12)
C4	0.0150 (15)	0.0132 (15)	0.0176 (16)	−0.0012 (12)	−0.0043 (12)	−0.0026 (12)
C5	0.0182 (16)	0.0178 (16)	0.0158 (16)	−0.0001 (13)	0.0012 (13)	−0.0071 (13)
C6	0.0133 (14)	0.0185 (16)	0.0110 (15)	0.0002 (12)	0.0014 (12)	−0.0019 (12)
C7	0.0291 (19)	0.0160 (17)	0.0263 (19)	−0.0043 (14)	0.0014 (15)	−0.0025 (14)
C8	0.0136 (14)	0.0121 (14)	0.0116 (15)	−0.0018 (11)	0.0014 (12)	−0.0007 (11)
C9	0.0159 (17)	0.051 (2)	0.0130 (17)	0.0006 (16)	−0.0027 (13)	−0.0080 (16)
C10	0.0116 (16)	0.052 (3)	0.0196 (18)	0.0007 (16)	−0.0013 (13)	−0.0082 (17)
C11	0.0187 (16)	0.0151 (15)	0.0136 (15)	−0.0004 (12)	0.0014 (12)	−0.0008 (12)
C12	0.0182 (16)	0.033 (2)	0.0107 (16)	−0.0011 (14)	−0.0025 (13)	0.0016 (14)
C13	0.0139 (15)	0.0313 (19)	0.0137 (16)	−0.0017 (14)	−0.0017 (12)	−0.0003 (14)
C14	0.0198 (17)	0.0288 (19)	0.0150 (16)	−0.0015 (14)	0.0024 (13)	0.0001 (14)
C15	0.0113 (14)	0.0171 (15)	0.0091 (14)	−0.0002 (12)	−0.0003 (11)	−0.0033 (12)
C16	0.0161 (15)	0.0174 (16)	0.0183 (16)	−0.0029 (12)	−0.0039 (13)	−0.0011 (13)
C17	0.0229 (17)	0.0164 (16)	0.0170 (16)	0.0029 (13)	−0.0032 (13)	−0.0033 (13)
C18	0.0151 (15)	0.0289 (18)	0.0105 (15)	0.0042 (13)	−0.0001 (12)	−0.0045 (13)
C19	0.0134 (15)	0.0254 (18)	0.0158 (16)	−0.0011 (13)	0.0012 (12)	−0.0026 (13)
C20	0.0161 (15)	0.0191 (16)	0.0153 (16)	−0.0019 (13)	0.0003 (12)	−0.0020 (13)
C21	0.0188 (17)	0.035 (2)	0.027 (2)	0.0091 (15)	−0.0022 (15)	−0.0020 (16)
C22	0.0166 (16)	0.0174 (16)	0.0123 (15)	−0.0021 (12)	−0.0022 (12)	−0.0043 (12)
C23	0.0130 (15)	0.0172 (16)	0.0191 (17)	−0.0014 (12)	−0.0008 (12)	−0.0049 (13)
C24	0.0105 (14)	0.0169 (16)	0.0169 (16)	0.0047 (12)	−0.0002 (12)	−0.0018 (12)
C25	0.0248 (18)	0.0175 (17)	0.0226 (18)	−0.0001 (14)	0.0013 (14)	0.0010 (14)
C26	0.0126 (14)	0.0067 (13)	0.0139 (15)	−0.0001 (11)	0.0000 (11)	0.0015 (11)
C27	0.0107 (14)	0.0164 (16)	0.0218 (17)	0.0017 (12)	−0.0004 (12)	−0.0046 (13)
C28	0.0143 (15)	0.0186 (17)	0.0184 (16)	−0.0022 (12)	−0.0020 (12)	−0.0046 (13)
C29	0.0188 (17)	0.0204 (17)	0.0231 (18)	−0.0023 (13)	−0.0044 (14)	−0.0137 (14)
C30	0.0168 (16)	0.0174 (16)	0.0194 (17)	0.0034 (13)	−0.0039 (13)	−0.0048 (13)
O1	0.0170 (12)	0.0357 (15)	0.0218 (13)	0.0083 (11)	−0.0028 (10)	−0.0093 (11)

O2	0.0264 (13)	0.0222 (13)	0.0249 (14)	−0.0046 (10)	0.0025 (11)	0.0052 (11)
O3	0.0231 (12)	0.0169 (12)	0.0133 (11)	−0.0057 (9)	−0.0018 (9)	−0.0018 (9)
O4	0.0220 (12)	0.0143 (11)	0.0154 (12)	0.0015 (9)	−0.0033 (9)	−0.0015 (9)
O5	0.0109 (10)	0.0222 (12)	0.0156 (11)	0.0009 (9)	−0.0002 (9)	−0.0064 (9)
O6	0.0108 (10)	0.0178 (11)	0.0147 (11)	0.0008 (9)	−0.0002 (8)	−0.0055 (9)
O7	0.0348 (15)	0.0190 (13)	0.0297 (15)	−0.0072 (11)	−0.0022 (12)	−0.0004 (11)
O8	0.0141 (13)	0.0394 (16)	0.0526 (19)	0.0008 (11)	−0.0028 (12)	−0.0274 (14)
O9	0.0373 (15)	0.0333 (15)	0.0224 (14)	0.0082 (12)	−0.0084 (12)	−0.0151 (12)
Os1	0.00957 (6)	0.01199 (7)	0.01086 (7)	−0.00047 (4)	0.00070 (4)	−0.00293 (4)
Os2	0.01044 (6)	0.01276 (7)	0.01335 (7)	−0.00026 (4)	−0.00131 (5)	−0.00473 (5)
P1	0.0100 (3)	0.0127 (4)	0.0104 (4)	−0.0016 (3)	0.0017 (3)	−0.0024 (3)

Geometric parameters (Å, º)

C1—C6	1.392 (4)	C18—C19	1.395 (5)
C1—C2	1.406 (4)	C18—C21	1.511 (5)
C1—P1	1.820 (3)	C19—C20	1.397 (5)
C2—C3	1.383 (4)	C19—H19	0.9500
C2—H2	0.9500	C20—H20	0.9500
C3—C4	1.398 (5)	C21—H21A	0.9800
C3—H3	0.9500	C21—H21B	0.9800
C4—C5	1.399 (5)	C21—H21C	0.9800
C4—C7	1.507 (5)	C22—O1	1.157 (4)
C5—C6	1.394 (5)	C22—Os1	1.858 (3)
C5—H5	0.9500	C23—O2	1.159 (4)
C6—H6	0.9500	C23—Os1	1.857 (3)
C7—H7A	0.9800	C24—O3	1.256 (4)
C7—H7B	0.9800	C24—O4	1.260 (4)
C7—H7C	0.9800	C24—C25	1.512 (5)
C8—C13	1.391 (4)	C25—H25A	0.9800
C8—C9	1.394 (5)	C25—H25B	0.9800
C8—P1	1.825 (3)	C25—H25C	0.9800
C9—C10	1.386 (5)	C26—O5	1.255 (4)
C9—H9	0.9500	C26—O6	1.269 (4)
C10—C11	1.385 (5)	C26—C27	1.502 (4)
C10—H10	0.9500	C27—H27A	0.9800
C11—C12	1.400 (5)	C27—H27B	0.9800
C11—C14	1.508 (5)	C27—H27C	0.9800
C12—C13	1.391 (5)	C28—O7	1.139 (4)
C12—H12	0.9500	C28—Os2	1.887 (3)
C13—H13	0.9500	C29—O8	1.148 (4)
C14—H14A	0.9800	C29—Os2	1.875 (3)
C14—H14B	0.9800	C30—O9	1.126 (4)
C14—H14C	0.9800	C30—Os2	1.979 (3)
C15—C20	1.395 (4)	O3—Os1	2.128 (2)
C15—C16	1.399 (4)	O4—Os2	2.122 (2)
C15—P1	1.834 (3)	O5—Os1	2.127 (2)
C16—C17	1.388 (5)	O6—Os2	2.124 (2)

C16—H16	0.9500	Os1—P1	2.4261 (10)
C17—C18	1.392 (5)	Os1—Os2	2.7624 (8)
C17—H17	0.9500		
C6—C1—C2	118.4 (3)	H21A—C21—H21B	109.5
C6—C1—P1	123.4 (2)	C18—C21—H21C	109.5
C2—C1—P1	118.3 (2)	H21A—C21—H21C	109.5
C3—C2—C1	120.6 (3)	H21B—C21—H21C	109.5
C3—C2—H2	119.7	O1—C22—Os1	176.5 (3)
C1—C2—H2	119.7	O2—C23—Os1	179.9 (4)
C2—C3—C4	121.2 (3)	O3—C24—O4	125.8 (3)
C2—C3—H3	119.4	O3—C24—C25	116.7 (3)
C4—C3—H3	119.4	O4—C24—C25	117.5 (3)
C3—C4—C5	118.1 (3)	C24—C25—H25A	109.5
C3—C4—C7	120.5 (3)	C24—C25—H25B	109.5
C5—C4—C7	121.3 (3)	H25A—C25—H25B	109.5
C6—C5—C4	120.8 (3)	C24—C25—H25C	109.5
C6—C5—H5	119.6	H25A—C25—H25C	109.5
C4—C5—H5	119.6	H25B—C25—H25C	109.5
C1—C6—C5	120.8 (3)	O5—C26—O6	125.1 (3)
C1—C6—H6	119.6	O5—C26—C27	118.1 (3)
C5—C6—H6	119.6	O6—C26—C27	116.7 (3)
C4—C7—H7A	109.5	C26—C27—H27A	109.5
C4—C7—H7B	109.5	C26—C27—H27B	109.5
H7A—C7—H7B	109.5	H27A—C27—H27B	109.5
C4—C7—H7C	109.5	C26—C27—H27C	109.5
H7A—C7—H7C	109.5	H27A—C27—H27C	109.5
H7B—C7—H7C	109.5	H27B—C27—H27C	109.5
C13—C8—C9	117.8 (3)	O7—C28—Os2	179.4 (3)
C13—C8—P1	122.5 (2)	O8—C29—Os2	179.5 (3)
C9—C8—P1	119.5 (2)	O9—C30—Os2	173.5 (3)
C10—C9—C8	121.0 (3)	C24—O3—Os1	125.3 (2)
C10—C9—H9	119.5	C24—O4—Os2	122.6 (2)
C8—C9—H9	119.5	C26—O5—Os1	125.1 (2)
C11—C10—C9	121.8 (3)	C26—O6—Os2	123.45 (19)
C11—C10—H10	119.1	C23—Os1—C22	90.83 (14)
C9—C10—H10	119.1	C23—Os1—O5	96.20 (12)
C10—C11—C12	117.2 (3)	C22—Os1—O5	172.36 (12)
C10—C11—C14	120.8 (3)	C23—Os1—O3	177.66 (12)
C12—C11—C14	122.0 (3)	C22—Os1—O3	89.97 (12)
C13—C12—C11	121.4 (3)	O5—Os1—O3	82.89 (9)
C13—C12—H12	119.3	C23—Os1—P1	90.97 (11)
C11—C12—H12	119.3	C22—Os1—P1	98.03 (10)
C12—C13—C8	120.9 (3)	O5—Os1—P1	84.88 (7)
C12—C13—H13	119.6	O3—Os1—P1	91.10 (7)
C8—C13—H13	119.6	C23—Os1—Os2	96.20 (10)
C11—C14—H14A	109.5	C22—Os1—Os2	94.14 (10)
C11—C14—H14B	109.5	O5—Os1—Os2	82.15 (6)

H14A—C14—H14B	109.5	O3—Os1—Os2	81.55 (7)
C11—C14—H14C	109.5	P1—Os1—Os2	165.78 (2)
H14A—C14—H14C	109.5	C29—Os2—C28	89.92 (15)
H14B—C14—H14C	109.5	C29—Os2—C30	94.50 (14)
C20—C15—C16	118.1 (3)	C28—Os2—C30	95.08 (14)
C20—C15—P1	123.0 (2)	C29—Os2—O4	93.53 (13)
C16—C15—P1	118.6 (2)	C28—Os2—O4	173.31 (11)
C17—C16—C15	120.7 (3)	C30—Os2—O4	90.37 (12)
C17—C16—H16	119.6	C29—Os2—O6	174.59 (11)
C15—C16—H16	119.6	C28—Os2—O6	92.36 (12)
C16—C17—C18	121.5 (3)	C30—Os2—O6	90.19 (11)
C16—C17—H17	119.3	O4—Os2—O6	83.72 (9)
C18—C17—H17	119.3	C29—Os2—Os1	91.62 (10)
C17—C18—C19	117.8 (3)	C28—Os2—Os1	90.16 (10)
C17—C18—C21	121.0 (3)	C30—Os2—Os1	171.95 (10)
C19—C18—C21	121.2 (3)	O4—Os2—Os1	84.02 (7)
C18—C19—C20	121.2 (3)	O6—Os2—Os1	83.47 (6)
C18—C19—H19	119.4	C1—P1—C8	103.95 (14)
C20—C19—H19	119.4	C1—P1—C15	104.43 (14)
C15—C20—C19	120.6 (3)	C8—P1—C15	104.85 (14)
C15—C20—H20	119.7	C1—P1—Os1	116.59 (11)
C19—C20—H20	119.7	C8—P1—Os1	115.81 (11)
C18—C21—H21A	109.5	C15—P1—Os1	109.99 (10)
C18—C21—H21B	109.5		

C6—C1—C2—C3	−1.1 (5)	P1—C15—C20—C19	171.4 (2)
P1—C1—C2—C3	177.7 (2)	C18—C19—C20—C15	0.6 (5)
C1—C2—C3—C4	1.0 (5)	O4—C24—O3—Os1	6.4 (5)
C2—C3—C4—C5	0.0 (5)	C25—C24—O3—Os1	−172.8 (2)
C2—C3—C4—C7	−178.3 (3)	O3—C24—O4—Os2	2.3 (4)
C3—C4—C5—C6	−0.9 (5)	C25—C24—O4—Os2	−178.5 (2)
C7—C4—C5—C6	177.3 (3)	O6—C26—O5—Os1	6.5 (4)
C2—C1—C6—C5	0.2 (5)	C27—C26—O5—Os1	−173.6 (2)
P1—C1—C6—C5	−178.6 (2)	O5—C26—O6—Os2	1.6 (4)
C4—C5—C6—C1	0.8 (5)	C27—C26—O6—Os2	−178.3 (2)
C13—C8—C9—C10	0.4 (6)	C6—C1—P1—C8	−0.2 (3)
P1—C8—C9—C10	174.8 (3)	C2—C1—P1—C8	−178.9 (2)
C8—C9—C10—C11	−1.1 (6)	C6—C1—P1—C15	109.5 (3)
C9—C10—C11—C12	1.4 (6)	C2—C1—P1—C15	−69.2 (3)
C9—C10—C11—C14	−179.0 (4)	C6—C1—P1—Os1	−128.9 (2)
C10—C11—C12—C13	−1.2 (5)	C2—C1—P1—Os1	52.3 (3)
C14—C11—C12—C13	179.3 (3)	C13—C8—P1—C1	82.1 (3)
C11—C12—C13—C8	0.6 (6)	C9—C8—P1—C1	−92.0 (3)
C9—C8—C13—C12	−0.2 (5)	C13—C8—P1—C15	−27.3 (3)
P1—C8—C13—C12	−174.4 (3)	C9—C8—P1—C15	158.6 (3)
C20—C15—C16—C17	1.5 (5)	C13—C8—P1—Os1	−148.7 (2)
P1—C15—C16—C17	−172.3 (3)	C9—C8—P1—Os1	37.2 (3)
C15—C16—C17—C18	0.7 (5)	C20—C15—P1—C1	6.1 (3)

C16—C17—C18—C19	−2.2 (5)	C16—C15—P1—C1	179.7 (3)
C16—C17—C18—C21	176.4 (3)	C20—C15—P1—C8	115.2 (3)
C17—C18—C19—C20	1.5 (5)	C16—C15—P1—C8	−71.3 (3)
C21—C18—C19—C20	−177.0 (3)	C20—C15—P1—Os1	−119.7 (3)
C16—C15—C20—C19	−2.2 (5)	C16—C15—P1—Os1	53.8 (3)

Bis(*μ*-acetato-1*κO*:2*κO′*)bis[tris(4-methylphenyl)phosphane]-1*κP*,2*κP*-bis(dicarbonylosmium)(*Os*—*Os*) *p*-xylene sesquisolvate (4@1.5C8H10)

Crystal data

$[Os_2(C_2H_3O_2)_2(C_{21}H_{21}P)_2(CO)_4]\cdot 1.5C_8H_{10}$
M_r = 1378.46
Triclinic, $P\overline{1}$
a = 12.81225 (16) Å
b = 14.8455 (2) Å
c = 16.6818 (2) Å
α = 98.1785 (11)°
β = 101.7904 (11)°
γ = 113.3846 (14)°
V = 2761.37 (7) Å^3
Z = 2
$F(000)$ = 1362
D_x = 1.658 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 23482 reflections
θ = 3.7–73.2°
μ = 9.55 mm^{-1}
T = 293 K
Rectangular plate, clear pale yellow
0.11 × 0.07 × 0.02 mm

Data collection

Rigaku SuperNova AtlasS2 CCD diffractometer
Radiation source: micro-focus sealed X-ray tube, SuperNova (Cu) X-ray Source
Mirror monochromator
Detector resolution: 5.2387 pixels mm^{-1}
ω scans
Absorption correction: gaussian (CrysAlis PRO; Rigaku OD, 2017)
T_{min} = 0.626, T_{max} = 1.000
49090 measured reflections
9768 independent reflections
8691 reflections with $I > 2\sigma(I)$
R_{int} = 0.066
θ_{max} = 66.6°, θ_{min} = 3.4°
h = −15→15
k = −17→17
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.068
S = 1.05
9768 reflections
678 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0326P)^2 + 0.5818P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.009
$\Delta\rho_{max}$ = 2.09 e Å^{-3}
$\Delta\rho_{min}$ = −1.11 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
Os1	0.52561 (2)	0.31103 (2)	0.17940 (2)	0.01350 (5)
Os2	0.72811 (2)	0.49183 (2)	0.22971 (2)	0.01374 (5)

P1	0.32377 (7)	0.18569 (6)	0.15247 (6)	0.01582 (17)
P2	0.87312 (7)	0.66724 (6)	0.28705 (5)	0.01529 (17)
O1	0.6466 (2)	0.2025 (2)	0.27154 (18)	0.0273 (6)
O2	0.5582 (2)	0.2355 (2)	0.01363 (17)	0.0262 (6)
O3	0.8908 (2)	0.3974 (2)	0.28500 (19)	0.0293 (6)
O4	0.7791 (2)	0.4713 (2)	0.06201 (18)	0.0302 (6)
O5	0.4957 (2)	0.37716 (19)	0.28926 (16)	0.0191 (5)
O6	0.6814 (2)	0.4997 (2)	0.34497 (16)	0.0229 (6)
O7	0.4501 (2)	0.39783 (19)	0.11990 (16)	0.0201 (5)
O8	0.6001 (2)	0.54765 (18)	0.19350 (17)	0.0210 (5)
C1	0.5991 (3)	0.2435 (3)	0.2357 (2)	0.0202 (8)
C2	0.5478 (3)	0.2634 (3)	0.0782 (2)	0.0191 (8)
C3	0.8295 (3)	0.4349 (3)	0.2647 (2)	0.0205 (8)
C4	0.7596 (3)	0.4799 (3)	0.1262 (2)	0.0204 (8)
C5	0.5784 (3)	0.4483 (3)	0.3484 (2)	0.0208 (8)
C6	0.5499 (4)	0.4710 (3)	0.4293 (3)	0.0281 (9)
H6A	0.471410	0.466933	0.416934	0.042*
H6B	0.606215	0.537999	0.461884	0.042*
H6C	0.553879	0.422658	0.460874	0.042*
C7	0.4981 (3)	0.4930 (3)	0.1434 (2)	0.0214 (8)
C8	0.4266 (4)	0.5450 (3)	0.1082 (3)	0.0332 (10)
H8A	0.423870	0.542089	0.049895	0.050*
H8B	0.462793	0.614544	0.139633	0.050*
H8C	0.347401	0.511789	0.112562	0.050*
C9	0.2214 (3)	0.2385 (3)	0.1172 (2)	0.0184 (7)
C10	0.1934 (3)	0.2931 (3)	0.1778 (2)	0.0198 (7)
H10	0.216126	0.292314	0.234216	0.024*
C11	0.1323 (3)	0.3483 (3)	0.1547 (2)	0.0224 (8)
H11	0.114982	0.384548	0.195946	0.027*
C12	0.0959 (3)	0.3506 (3)	0.0702 (3)	0.0229 (8)
C13	0.1244 (3)	0.2967 (3)	0.0105 (3)	0.0268 (8)
H13	0.101210	0.297283	−0.045899	0.032*
C14	0.1866 (3)	0.2418 (3)	0.0330 (2)	0.0233 (8)
H14	0.205414	0.206952	−0.008293	0.028*
C15	0.0313 (4)	0.4125 (3)	0.0454 (3)	0.0313 (9)
H15A	−0.005020	0.424502	0.088157	0.047*
H15B	−0.028828	0.376251	−0.007556	0.047*
H15C	0.086583	0.476050	0.039950	0.047*
C16	0.2803 (3)	0.0689 (3)	0.0732 (2)	0.0183 (7)
C17	0.1659 (3)	0.0121 (3)	0.0176 (2)	0.0228 (8)
H17	0.108135	0.034979	0.018611	0.027*
C18	0.1379 (3)	−0.0777 (3)	−0.0388 (2)	0.0237 (8)
H18	0.061579	−0.114091	−0.075470	0.028*
C19	0.2219 (3)	−0.1146 (3)	−0.0416 (2)	0.0197 (7)
C20	0.3357 (3)	−0.0581 (3)	0.0153 (2)	0.0208 (7)
H20	0.393119	−0.081477	0.015326	0.025*
C21	0.3637 (3)	0.0315 (3)	0.0712 (2)	0.0210 (8)
H21	0.439844	0.067623	0.108184	0.025*

C22	0.1942 (3)	−0.2102 (3)	−0.1050 (2)	0.0250 (8)
H22A	0.245412	−0.194378	−0.140910	0.037*
H22B	0.112994	−0.239125	−0.138782	0.037*
H22C	0.206713	−0.257824	−0.075699	0.037*
C23	0.2753 (3)	0.1389 (3)	0.2410 (2)	0.0183 (7)
C24	0.1564 (3)	0.0729 (3)	0.2272 (3)	0.0235 (8)
H24	0.104064	0.050648	0.173139	0.028*
C25	0.1153 (3)	0.0399 (3)	0.2936 (3)	0.0232 (8)
H25	0.035691	−0.004162	0.283412	0.028*
C26	0.1916 (3)	0.0719 (3)	0.3750 (3)	0.0229 (8)
C27	0.3100 (3)	0.1376 (3)	0.3879 (3)	0.0264 (8)
H27	0.362345	0.160071	0.442027	0.032*
C28	0.3521 (3)	0.1706 (3)	0.3214 (3)	0.0245 (8)
H28	0.431883	0.213904	0.331386	0.029*
C29	0.1465 (4)	0.0355 (3)	0.4471 (3)	0.0323 (9)
H29A	0.080771	0.050251	0.450432	0.048*
H29B	0.208936	0.069394	0.499248	0.048*
H29C	0.121051	−0.036301	0.437346	0.048*
C30	0.9286 (3)	0.7089 (3)	0.4023 (2)	0.0178 (7)
C31	0.8474 (3)	0.7050 (3)	0.4485 (2)	0.0198 (7)
H31	0.767888	0.683094	0.420013	0.024*
C32	0.8847 (3)	0.7333 (3)	0.5358 (2)	0.0207 (7)
H32	0.829639	0.730875	0.565175	0.025*
C33	1.0028 (3)	0.7654 (3)	0.5810 (2)	0.0218 (8)
C34	1.0829 (3)	0.7684 (3)	0.5350 (2)	0.0228 (8)
H34	1.161987	0.789112	0.563842	0.027*
C35	1.0477 (3)	0.7413 (3)	0.4474 (2)	0.0206 (7)
H35	1.103286	0.744667	0.418307	0.025*
C36	1.0416 (4)	0.7940 (3)	0.6762 (2)	0.0286 (9)
H36A	1.038792	0.856629	0.696442	0.043*
H36B	1.121316	0.801975	0.696327	0.043*
H36C	0.989527	0.741690	0.696652	0.043*
C37	1.0046 (3)	0.6985 (3)	0.2496 (2)	0.0173 (7)
C38	1.0630 (3)	0.6365 (3)	0.2544 (3)	0.0235 (8)
H38	1.038309	0.583498	0.280537	0.028*
C39	1.1576 (3)	0.6532 (3)	0.2204 (3)	0.0244 (8)
H39	1.196303	0.612016	0.225191	0.029*
C40	1.1954 (3)	0.7304 (3)	0.1796 (3)	0.0258 (8)
C41	1.1394 (3)	0.7934 (3)	0.1769 (3)	0.0270 (9)
H41	1.164746	0.846531	0.151005	0.032*
C42	1.0463 (3)	0.7788 (3)	0.2120 (3)	0.0244 (8)
H42	1.011337	0.822979	0.210336	0.029*
C43	1.2929 (4)	0.7427 (4)	0.1380 (3)	0.0337 (10)
H43A	1.357108	0.738327	0.175825	0.050*
H43B	1.321035	0.807465	0.124746	0.050*
H43C	1.262384	0.689978	0.086924	0.050*
C44	0.8176 (3)	0.7585 (3)	0.2589 (2)	0.0170 (7)
C45	0.8316 (3)	0.8426 (3)	0.3172 (2)	0.0195 (7)

H45	0.874379	0.856310	0.373248	0.023*
C46	0.7829 (3)	0.9061 (3)	0.2928 (3)	0.0237 (8)
H46	0.793734	0.961822	0.332987	0.028*
C47	0.7182 (3)	0.8885 (3)	0.2098 (3)	0.0240 (8)
C48	0.7060 (3)	0.8052 (3)	0.1513 (3)	0.0248 (8)
H48	0.664560	0.792533	0.095008	0.030*
C49	0.7538 (3)	0.7410 (3)	0.1748 (2)	0.0211 (7)
H49	0.743559	0.685698	0.134480	0.025*
C50	0.6617 (4)	0.9549 (3)	0.1835 (3)	0.0347 (10)
H50A	0.576957	0.917897	0.169453	0.052*
H50B	0.683391	0.975972	0.135111	0.052*
H50C	0.688619	1.013497	0.229212	0.052*
C51	0.4260 (4)	0.6645 (3)	0.3715 (3)	0.0303 (9)
C52	0.4041 (3)	0.7228 (3)	0.4328 (3)	0.0308 (9)
H52	0.453454	0.791965	0.452394	0.037*
C53	0.3088 (4)	0.6788 (3)	0.4654 (3)	0.0298 (9)
H53	0.296385	0.719046	0.507002	0.036*
C54	0.2323 (3)	0.5762 (3)	0.4369 (3)	0.0269 (9)
C55	0.2548 (4)	0.5190 (3)	0.3750 (3)	0.0285 (9)
H55	0.204875	0.449997	0.354793	0.034*
C56	0.3493 (4)	0.5618 (3)	0.3425 (3)	0.0302 (9)
H56	0.361549	0.521472	0.300929	0.036*
C57	0.5316 (4)	0.7132 (4)	0.3385 (3)	0.0383 (10)
H57A	0.546104	0.661414	0.308249	0.057*
H57B	0.600382	0.756331	0.385135	0.057*
H57C	0.515246	0.752699	0.301284	0.057*
C58	0.1317 (4)	0.5287 (3)	0.4742 (3)	0.0360 (10)
H58A	0.063317	0.478694	0.430344	0.054*
H58B	0.112728	0.580063	0.499867	0.054*
H58C	0.155174	0.497014	0.516227	0.054*
C59	0.6146 (4)	0.0436 (4)	0.5492 (4)	0.0483 (14)
H59	0.693393	0.073771	0.581970	0.058*
C60	0.5899 (5)	0.0302 (3)	0.4632 (4)	0.0484 (14)
H60	0.652302	0.051126	0.439229	0.058*
C61	0.4750 (5)	−0.0134 (3)	0.4113 (4)	0.0434 (12)
C62	0.4498 (7)	−0.0286 (5)	0.3172 (4)	0.0687 (18)
H62A	0.370174	−0.038161	0.293340	0.103*
H62B	0.458306	−0.087231	0.293593	0.103*
H62C	0.504674	0.029967	0.304633	0.103*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Os1	0.01122 (8)	0.01568 (8)	0.01396 (9)	0.00621 (6)	0.00354 (6)	0.00397 (6)
Os2	0.01186 (8)	0.01575 (8)	0.01351 (9)	0.00615 (6)	0.00287 (6)	0.00414 (6)
P1	0.0121 (4)	0.0176 (4)	0.0184 (4)	0.0067 (3)	0.0049 (3)	0.0049 (3)
P2	0.0138 (4)	0.0166 (4)	0.0154 (4)	0.0067 (3)	0.0037 (3)	0.0043 (3)
O1	0.0222 (13)	0.0301 (14)	0.0351 (16)	0.0158 (12)	0.0052 (11)	0.0158 (12)

O2	0.0272 (14)	0.0324 (14)	0.0188 (15)	0.0140 (12)	0.0083 (11)	0.0012 (11)
O3	0.0202 (13)	0.0274 (14)	0.0380 (17)	0.0134 (12)	−0.0026 (11)	0.0088 (12)
O4	0.0268 (14)	0.0426 (17)	0.0212 (16)	0.0132 (13)	0.0104 (11)	0.0090 (12)
O5	0.0178 (12)	0.0248 (13)	0.0164 (13)	0.0099 (10)	0.0081 (10)	0.0040 (10)
O6	0.0234 (13)	0.0257 (13)	0.0140 (13)	0.0054 (11)	0.0074 (10)	0.0013 (10)
O7	0.0134 (11)	0.0230 (13)	0.0245 (14)	0.0094 (10)	0.0022 (10)	0.0080 (10)
O8	0.0157 (12)	0.0193 (12)	0.0271 (14)	0.0084 (10)	0.0037 (10)	0.0050 (10)
C1	0.0131 (16)	0.0185 (17)	0.0234 (19)	0.0010 (14)	0.0085 (14)	0.0019 (14)
C2	0.0126 (16)	0.0211 (17)	0.023 (2)	0.0071 (14)	0.0014 (14)	0.0098 (15)
C3	0.0174 (17)	0.0176 (16)	0.0191 (19)	0.0020 (14)	0.0032 (14)	0.0025 (14)
C4	0.0156 (17)	0.0235 (18)	0.017 (2)	0.0046 (14)	0.0029 (14)	0.0058 (14)
C5	0.0222 (19)	0.0252 (18)	0.023 (2)	0.0146 (16)	0.0113 (15)	0.0100 (15)
C6	0.031 (2)	0.037 (2)	0.020 (2)	0.0171 (18)	0.0108 (16)	0.0061 (16)
C7	0.0218 (18)	0.0230 (18)	0.0199 (19)	0.0101 (15)	0.0055 (15)	0.0066 (14)
C8	0.023 (2)	0.027 (2)	0.050 (3)	0.0144 (17)	0.0009 (18)	0.0137 (19)
C9	0.0133 (16)	0.0208 (17)	0.0197 (19)	0.0060 (13)	0.0041 (13)	0.0056 (14)
C10	0.0162 (16)	0.0210 (17)	0.0190 (18)	0.0073 (14)	0.0005 (13)	0.0048 (14)
C11	0.0190 (17)	0.0219 (17)	0.026 (2)	0.0100 (15)	0.0059 (15)	0.0037 (15)
C12	0.0163 (17)	0.0267 (18)	0.031 (2)	0.0108 (15)	0.0076 (15)	0.0160 (16)
C13	0.0201 (18)	0.039 (2)	0.028 (2)	0.0150 (17)	0.0090 (15)	0.0173 (17)
C14	0.0200 (18)	0.032 (2)	0.021 (2)	0.0131 (16)	0.0069 (15)	0.0083 (15)
C15	0.028 (2)	0.034 (2)	0.040 (3)	0.0198 (18)	0.0094 (18)	0.0157 (19)
C16	0.0146 (16)	0.0218 (17)	0.0185 (18)	0.0073 (14)	0.0050 (13)	0.0068 (14)
C17	0.0167 (18)	0.0278 (19)	0.025 (2)	0.0114 (15)	0.0062 (15)	0.0060 (15)
C18	0.0126 (17)	0.0299 (19)	0.023 (2)	0.0060 (15)	0.0019 (14)	0.0045 (15)
C19	0.0199 (17)	0.0196 (17)	0.0193 (19)	0.0071 (14)	0.0071 (14)	0.0068 (14)
C20	0.0175 (17)	0.0230 (18)	0.024 (2)	0.0106 (14)	0.0063 (14)	0.0072 (15)
C21	0.0141 (16)	0.0229 (18)	0.0199 (19)	0.0044 (14)	0.0011 (14)	0.0042 (14)
C22	0.0226 (18)	0.0245 (18)	0.023 (2)	0.0087 (15)	0.0041 (15)	0.0003 (15)
C23	0.0177 (17)	0.0211 (17)	0.0208 (19)	0.0108 (14)	0.0093 (14)	0.0072 (14)
C24	0.0209 (18)	0.0240 (18)	0.024 (2)	0.0088 (15)	0.0070 (15)	0.0043 (15)
C25	0.0166 (17)	0.0230 (18)	0.031 (2)	0.0074 (14)	0.0119 (15)	0.0072 (15)
C26	0.0280 (19)	0.0201 (17)	0.029 (2)	0.0137 (15)	0.0144 (16)	0.0113 (15)
C27	0.026 (2)	0.0286 (19)	0.023 (2)	0.0101 (16)	0.0044 (16)	0.0095 (16)
C28	0.0196 (18)	0.0251 (18)	0.025 (2)	0.0059 (15)	0.0037 (15)	0.0106 (15)
C29	0.037 (2)	0.031 (2)	0.031 (2)	0.0121 (18)	0.0162 (18)	0.0127 (18)
C30	0.0192 (17)	0.0187 (16)	0.0175 (18)	0.0107 (14)	0.0031 (14)	0.0067 (13)
C31	0.0164 (17)	0.0204 (17)	0.0229 (19)	0.0090 (14)	0.0036 (14)	0.0065 (14)
C32	0.0233 (18)	0.0190 (17)	0.022 (2)	0.0100 (14)	0.0089 (15)	0.0066 (14)
C33	0.030 (2)	0.0171 (16)	0.0207 (19)	0.0124 (15)	0.0058 (15)	0.0062 (14)
C34	0.0185 (17)	0.0223 (17)	0.023 (2)	0.0081 (14)	−0.0007 (14)	0.0032 (14)
C35	0.0177 (17)	0.0231 (17)	0.0209 (19)	0.0103 (14)	0.0035 (14)	0.0045 (14)
C36	0.033 (2)	0.032 (2)	0.022 (2)	0.0176 (18)	0.0048 (16)	0.0047 (16)
C37	0.0149 (16)	0.0175 (16)	0.0180 (18)	0.0073 (13)	0.0035 (13)	0.0016 (13)
C38	0.0202 (18)	0.0263 (19)	0.029 (2)	0.0111 (15)	0.0103 (15)	0.0133 (16)
C39	0.0199 (18)	0.0278 (19)	0.030 (2)	0.0145 (15)	0.0080 (15)	0.0077 (16)
C40	0.0209 (18)	0.036 (2)	0.024 (2)	0.0134 (16)	0.0091 (15)	0.0105 (16)
C41	0.0228 (19)	0.033 (2)	0.037 (2)	0.0153 (16)	0.0159 (17)	0.0217 (18)

C42	0.0215 (18)	0.0241 (18)	0.033 (2)	0.0122 (15)	0.0097 (16)	0.0127 (16)
C43	0.029 (2)	0.047 (2)	0.037 (2)	0.0218 (19)	0.0172 (18)	0.019 (2)
C44	0.0122 (15)	0.0193 (16)	0.0219 (19)	0.0075 (13)	0.0068 (13)	0.0079 (14)
C45	0.0166 (17)	0.0223 (17)	0.0213 (19)	0.0086 (14)	0.0073 (14)	0.0077 (14)
C46	0.0250 (19)	0.0189 (17)	0.031 (2)	0.0104 (15)	0.0122 (16)	0.0091 (15)
C47	0.0186 (18)	0.0278 (19)	0.032 (2)	0.0110 (15)	0.0123 (15)	0.0156 (16)
C48	0.0199 (18)	0.030 (2)	0.025 (2)	0.0095 (15)	0.0067 (15)	0.0146 (16)
C49	0.0203 (18)	0.0247 (18)	0.0206 (19)	0.0104 (15)	0.0081 (14)	0.0075 (14)
C50	0.035 (2)	0.037 (2)	0.046 (3)	0.0227 (19)	0.015 (2)	0.024 (2)
C51	0.024 (2)	0.035 (2)	0.035 (2)	0.0179 (17)	0.0029 (17)	0.0131 (18)
C52	0.0186 (19)	0.027 (2)	0.042 (3)	0.0088 (16)	0.0020 (17)	0.0077 (18)
C53	0.024 (2)	0.030 (2)	0.040 (3)	0.0174 (17)	0.0077 (17)	0.0066 (18)
C54	0.0182 (18)	0.031 (2)	0.032 (2)	0.0139 (16)	−0.0015 (15)	0.0118 (17)
C55	0.028 (2)	0.0253 (19)	0.028 (2)	0.0117 (16)	−0.0011 (16)	0.0077 (16)
C56	0.034 (2)	0.031 (2)	0.025 (2)	0.0188 (18)	0.0002 (17)	0.0051 (17)
C57	0.029 (2)	0.045 (3)	0.045 (3)	0.019 (2)	0.013 (2)	0.015 (2)
C58	0.027 (2)	0.033 (2)	0.049 (3)	0.0144 (18)	0.0086 (19)	0.017 (2)
C59	0.032 (2)	0.028 (2)	0.090 (5)	0.0139 (19)	0.025 (3)	0.018 (2)
C60	0.049 (3)	0.027 (2)	0.088 (4)	0.020 (2)	0.047 (3)	0.024 (2)
C61	0.050 (3)	0.027 (2)	0.062 (3)	0.019 (2)	0.027 (2)	0.014 (2)
C62	0.094 (5)	0.043 (3)	0.075 (5)	0.029 (3)	0.036 (4)	0.014 (3)

Geometric parameters (Å, º)

Os1—Os2	2.7534 (2)	C29—H29A	0.9600
Os1—P1	2.4123 (8)	C29—H29B	0.9600
Os1—O5	2.132 (2)	C29—H29C	0.9600
Os1—O7	2.131 (2)	C30—C31	1.402 (5)
Os1—C1	1.852 (4)	C30—C35	1.404 (5)
Os1—C2	1.857 (4)	C31—H31	0.9300
Os2—P2	2.4233 (8)	C31—C32	1.382 (5)
Os2—O6	2.129 (3)	C32—H32	0.9300
Os2—O8	2.135 (2)	C32—C33	1.395 (5)
Os2—C3	1.857 (4)	C33—C34	1.391 (6)
Os2—C4	1.853 (4)	C33—C36	1.507 (5)
P1—C9	1.821 (4)	C34—H34	0.9300
P1—C16	1.829 (4)	C34—C35	1.386 (6)
P1—C23	1.836 (4)	C35—H35	0.9300
P2—C30	1.828 (4)	C36—H36A	0.9600
P2—C37	1.826 (4)	C36—H36B	0.9600
P2—C44	1.836 (4)	C36—H36C	0.9600
O1—C1	1.161 (5)	C37—C38	1.399 (5)
O2—C2	1.151 (5)	C37—C42	1.395 (5)
O3—C3	1.153 (5)	C38—H38	0.9300
O4—C4	1.149 (5)	C38—C39	1.390 (6)
O5—C5	1.265 (5)	C39—H39	0.9300
O6—C5	1.253 (5)	C39—C40	1.389 (6)
O7—C7	1.257 (5)	C40—C41	1.386 (6)

O8—C7	1.259 (5)	C40—C43	1.511 (6)
C5—C6	1.495 (5)	C41—H41	0.9300
C6—H6A	0.9600	C41—C42	1.390 (6)
C6—H6B	0.9600	C42—H42	0.9300
C6—H6C	0.9600	C43—H43A	0.9600
C7—C8	1.501 (5)	C43—H43B	0.9600
C8—H8A	0.9600	C43—H43C	0.9600
C8—H8B	0.9600	C44—C45	1.394 (5)
C8—H8C	0.9600	C44—C49	1.400 (5)
C9—C10	1.398 (5)	C45—H45	0.9300
C9—C14	1.398 (5)	C45—C46	1.384 (5)
C10—H10	0.9300	C46—H46	0.9300
C10—C11	1.381 (5)	C46—C47	1.390 (6)
C11—H11	0.9300	C47—C48	1.395 (6)
C11—C12	1.401 (6)	C47—C50	1.499 (5)
C12—C13	1.385 (6)	C48—H48	0.9300
C12—C15	1.507 (5)	C48—C49	1.382 (5)
C13—H13	0.9300	C49—H49	0.9300
C13—C14	1.385 (6)	C50—H50A	0.9600
C14—H14	0.9300	C50—H50B	0.9600
C15—H15A	0.9600	C50—H50C	0.9600
C15—H15B	0.9600	C51—C52	1.391 (7)
C15—H15C	0.9600	C51—C56	1.389 (6)
C16—C17	1.400 (5)	C51—C57	1.516 (6)
C16—C21	1.389 (5)	C52—H52	0.9300
C17—H17	0.9300	C52—C53	1.397 (6)
C17—C18	1.385 (6)	C53—H53	0.9300
C18—H18	0.9300	C53—C54	1.388 (6)
C18—C19	1.393 (5)	C54—C55	1.392 (6)
C19—C20	1.403 (5)	C54—C58	1.509 (6)
C19—C22	1.510 (5)	C55—H55	0.9300
C20—H20	0.9300	C55—C56	1.386 (6)
C20—C21	1.377 (5)	C56—H56	0.9300
C21—H21	0.9300	C57—H57A	0.9600
C22—H22A	0.9600	C57—H57B	0.9600
C22—H22B	0.9600	C57—H57C	0.9600
C22—H22C	0.9600	C58—H58A	0.9600
C23—C24	1.393 (5)	C58—H58B	0.9600
C23—C28	1.380 (5)	C58—H58C	0.9600
C24—H24	0.9300	C59—H59	0.9300
C24—C25	1.389 (5)	C59—C60	1.371 (9)
C25—H25	0.9300	C59—C61^{i}	1.392 (7)
C25—C26	1.390 (6)	C60—H60	0.9300
C26—C27	1.389 (5)	C60—C61	1.377 (8)
C26—C29	1.515 (5)	C61—C62	1.501 (9)
C27—H27	0.9300	C62—H62A	0.9600
C27—C28	1.397 (6)	C62—H62B	0.9600
C28—H28	0.9300	C62—H62C	0.9600

P1—Os1—Os2	162.49 (2)	C26—C27—C28	121.4 (4)
O5—Os1—Os2	81.65 (7)	C28—C27—H27	119.3
O5—Os1—P1	83.89 (7)	C23—C28—C27	120.0 (4)
O7—Os1—Os2	82.78 (6)	C23—C28—H28	120.0
O7—Os1—P1	85.94 (7)	C27—C28—H28	120.0
O7—Os1—O5	83.98 (10)	C26—C29—H29A	109.5
C1—Os1—Os2	93.70 (10)	C26—C29—H29B	109.5
C1—Os1—P1	97.17 (10)	C26—C29—H29C	109.5
C1—Os1—O5	94.24 (13)	H29A—C29—H29B	109.5
C1—Os1—O7	176.25 (12)	H29A—C29—H29C	109.5
C1—Os1—C2	91.78 (16)	H29B—C29—H29C	109.5
C2—Os1—Os2	96.48 (10)	C31—C30—P2	118.5 (3)
C2—Os1—P1	96.86 (11)	C31—C30—C35	118.2 (3)
C2—Os1—O5	173.80 (12)	C35—C30—P2	123.3 (3)
C2—Os1—O7	89.93 (13)	C30—C31—H31	119.7
P2—Os2—Os1	165.16 (2)	C32—C31—C30	120.5 (3)
O6—Os2—Os1	82.41 (7)	C32—C31—H31	119.7
O6—Os2—P2	87.53 (7)	C31—C32—H32	119.2
O6—Os2—O8	83.13 (11)	C31—C32—C33	121.6 (4)
O8—Os2—Os1	81.09 (7)	C33—C32—H32	119.2
O8—Os2—P2	86.92 (7)	C32—C33—C36	121.0 (4)
C3—Os2—Os1	94.75 (10)	C34—C33—C32	117.7 (4)
C3—Os2—P2	96.72 (11)	C34—C33—C36	121.3 (4)
C3—Os2—O6	93.37 (14)	C33—C34—H34	119.1
C3—Os2—O8	174.87 (13)	C35—C34—C33	121.7 (3)
C4—Os2—Os1	94.51 (11)	C35—C34—H34	119.1
C4—Os2—P2	95.24 (11)	C30—C35—H35	119.9
C4—Os2—O6	176.67 (12)	C34—C35—C30	120.3 (4)
C4—Os2—O8	95.15 (14)	C34—C35—H35	119.9
C4—Os2—C3	88.15 (16)	C33—C36—H36A	109.5
C9—P1—Os1	109.92 (11)	C33—C36—H36B	109.5
C9—P1—C16	107.47 (16)	C33—C36—H36C	109.5
C9—P1—C23	101.55 (16)	H36A—C36—H36B	109.5
C16—P1—Os1	114.91 (12)	H36A—C36—H36C	109.5
C16—P1—C23	102.87 (16)	H36B—C36—H36C	109.5
C23—P1—Os1	118.80 (12)	C38—C37—P2	119.1 (3)
C30—P2—Os2	115.60 (12)	C42—C37—P2	122.8 (3)
C30—P2—C44	103.11 (16)	C42—C37—C38	118.0 (3)
C37—P2—Os2	113.05 (11)	C37—C38—H38	119.7
C37—P2—C30	105.73 (16)	C39—C38—C37	120.6 (4)
C37—P2—C44	104.30 (16)	C39—C38—H38	119.7
C44—P2—Os2	113.87 (11)	C38—C39—H39	119.4
C5—O5—Os1	122.6 (2)	C40—C39—C38	121.2 (4)
C5—O6—Os2	122.3 (2)	C40—C39—H39	119.4
C7—O7—Os1	121.3 (2)	C39—C40—C43	120.2 (4)
C7—O8—Os2	123.0 (2)	C41—C40—C39	118.0 (4)
O1—C1—Os1	179.0 (3)	C41—C40—C43	121.8 (4)

O2—C2—Os1	176.8 (3)	C40—C41—H41	119.3
O3—C3—Os2	178.4 (3)	C40—C41—C42	121.4 (4)
O4—C4—Os2	179.1 (3)	C42—C41—H41	119.3
O5—C5—C6	116.4 (3)	C37—C42—H42	119.7
O6—C5—O5	125.6 (3)	C41—C42—C37	120.7 (4)
O6—C5—C6	118.0 (3)	C41—C42—H42	119.7
C5—C6—H6A	109.5	C40—C43—H43A	109.5
C5—C6—H6B	109.5	C40—C43—H43B	109.5
C5—C6—H6C	109.5	C40—C43—H43C	109.5
H6A—C6—H6B	109.5	H43A—C43—H43B	109.5
H6A—C6—H6C	109.5	H43A—C43—H43C	109.5
H6B—C6—H6C	109.5	H43B—C43—H43C	109.5
O7—C7—O8	125.7 (3)	C45—C44—P2	123.6 (3)
O7—C7—C8	116.6 (3)	C45—C44—C49	117.8 (3)
O8—C7—C8	117.7 (3)	C49—C44—P2	118.5 (3)
C7—C8—H8A	109.5	C44—C45—H45	119.5
C7—C8—H8B	109.5	C46—C45—C44	121.0 (3)
C7—C8—H8C	109.5	C46—C45—H45	119.5
H8A—C8—H8B	109.5	C45—C46—H46	119.3
H8A—C8—H8C	109.5	C45—C46—C47	121.5 (4)
H8B—C8—H8C	109.5	C47—C46—H46	119.3
C10—C9—P1	118.8 (3)	C46—C47—C48	117.4 (3)
C10—C9—C14	118.3 (3)	C46—C47—C50	121.8 (4)
C14—C9—P1	121.9 (3)	C48—C47—C50	120.8 (4)
C9—C10—H10	119.7	C47—C48—H48	119.2
C11—C10—C9	120.6 (3)	C49—C48—C47	121.7 (4)
C11—C10—H10	119.7	C49—C48—H48	119.2
C10—C11—H11	119.4	C44—C49—H49	119.7
C10—C11—C12	121.2 (4)	C48—C49—C44	120.6 (4)
C12—C11—H11	119.4	C48—C49—H49	119.7
C11—C12—C15	121.0 (4)	C47—C50—H50A	109.5
C13—C12—C11	117.8 (3)	C47—C50—H50B	109.5
C13—C12—C15	121.1 (4)	C47—C50—H50C	109.5
C12—C13—H13	119.2	H50A—C50—H50B	109.5
C12—C13—C14	121.5 (4)	H50A—C50—H50C	109.5
C14—C13—H13	119.2	H50B—C50—H50C	109.5
C9—C14—H14	119.8	C52—C51—C57	120.0 (4)
C13—C14—C9	120.5 (4)	C56—C51—C52	118.2 (4)
C13—C14—H14	119.8	C56—C51—C57	121.8 (4)
C12—C15—H15A	109.5	C51—C52—H52	119.6
C12—C15—H15B	109.5	C51—C52—C53	120.8 (4)
C12—C15—H15C	109.5	C53—C52—H52	119.6
H15A—C15—H15B	109.5	C52—C53—H53	119.4
H15A—C15—H15C	109.5	C54—C53—C52	121.2 (4)
H15B—C15—H15C	109.5	C54—C53—H53	119.4
C17—C16—P1	123.6 (3)	C53—C54—C55	117.3 (4)
C21—C16—P1	118.1 (3)	C53—C54—C58	120.9 (4)
C21—C16—C17	118.2 (3)	C55—C54—C58	121.8 (4)

C16—C17—H17	119.7	C54—C55—H55	119.0
C18—C17—C16	120.6 (3)	C56—C55—C54	121.9 (4)
C18—C17—H17	119.7	C56—C55—H55	119.0
C17—C18—H18	119.4	C51—C56—H56	119.7
C17—C18—C19	121.3 (3)	C55—C56—C51	120.6 (4)
C19—C18—H18	119.4	C55—C56—H56	119.7
C18—C19—C20	117.7 (3)	C51—C57—H57A	109.5
C18—C19—C22	122.0 (3)	C51—C57—H57B	109.5
C20—C19—C22	120.3 (3)	C51—C57—H57C	109.5
C19—C20—H20	119.5	H57A—C57—H57B	109.5
C21—C20—C19	121.0 (3)	H57A—C57—H57C	109.5
C21—C20—H20	119.5	H57B—C57—H57C	109.5
C16—C21—H21	119.4	C54—C58—H58A	109.5
C20—C21—C16	121.2 (3)	C54—C58—H58B	109.5
C20—C21—H21	119.4	C54—C58—H58C	109.5
C19—C22—H22A	109.5	H58A—C58—H58B	109.5
C19—C22—H22B	109.5	H58A—C58—H58C	109.5
C19—C22—H22C	109.5	H58B—C58—H58C	109.5
H22A—C22—H22B	109.5	C60—C59—H59	119.1
H22A—C22—H22C	109.5	C60—C59—C61^{i}	121.8 (5)
H22B—C22—H22C	109.5	C61^{i}—C59—H59	119.1
C24—C23—P1	119.0 (3)	C59—C60—H60	119.1
C28—C23—P1	121.9 (3)	C59—C60—C61	121.8 (5)
C28—C23—C24	119.1 (3)	C61—C60—H60	119.1
C23—C24—H24	119.7	C59^{i}—C61—C62	122.6 (6)
C25—C24—C23	120.6 (4)	C60—C61—C59^{i}	116.4 (5)
C25—C24—H24	119.7	C60—C61—C62	121.0 (5)
C24—C25—H25	119.6	C61—C62—H62A	109.5
C24—C25—C26	120.9 (3)	C61—C62—H62B	109.5
C26—C25—H25	119.6	C61—C62—H62C	109.5
C25—C26—C29	120.6 (4)	H62A—C62—H62B	109.5
C27—C26—C25	118.0 (3)	H62A—C62—H62C	109.5
C27—C26—C29	121.3 (4)	H62B—C62—H62C	109.5
C26—C27—H27	119.3		

Os1—P1—C9—C10	−85.8 (3)	C23—P1—C16—C21	−93.0 (3)
Os1—P1—C9—C14	82.6 (3)	C23—C24—C25—C26	0.0 (6)
Os1—P1—C16—C17	−146.1 (3)	C24—C23—C28—C27	−0.9 (6)
Os1—P1—C16—C21	37.6 (3)	C24—C25—C26—C27	−0.1 (6)
Os1—P1—C23—C24	174.7 (2)	C24—C25—C26—C29	−179.8 (4)
Os1—P1—C23—C28	−2.4 (4)	C25—C26—C27—C28	−0.3 (6)
Os1—O5—C5—O6	12.5 (5)	C26—C27—C28—C23	0.8 (6)
Os1—O5—C5—C6	−166.5 (2)	C28—C23—C24—C25	0.5 (6)
Os1—O7—C7—O8	10.6 (5)	C29—C26—C27—C28	179.4 (4)
Os1—O7—C7—C8	−169.2 (3)	C30—P2—C37—C38	77.4 (3)
Os2—P2—C30—C31	−61.2 (3)	C30—P2—C37—C42	−106.2 (3)
Os2—P2—C30—C35	116.4 (3)	C30—P2—C44—C45	5.9 (3)
Os2—P2—C37—C38	−50.0 (3)	C30—P2—C44—C49	−171.6 (3)

Os2—P2—C37—C42	126.4 (3)	C30—C31—C32—C33	−0.8 (5)
Os2—P2—C44—C45	131.9 (3)	C31—C30—C35—C34	0.0 (5)
Os2—P2—C44—C49	−45.6 (3)	C31—C32—C33—C34	0.2 (5)
Os2—O6—C5—O5	10.4 (5)	C31—C32—C33—C36	−178.7 (3)
Os2—O6—C5—C6	−170.6 (3)	C32—C33—C34—C35	0.5 (5)
Os2—O8—C7—O7	13.3 (6)	C33—C34—C35—C30	−0.6 (6)
Os2—O8—C7—C8	−166.9 (3)	C35—C30—C31—C32	0.6 (5)
P1—C9—C10—C11	169.3 (3)	C36—C33—C34—C35	179.4 (4)
P1—C9—C14—C13	−169.5 (3)	C37—P2—C30—C31	172.9 (3)
P1—C16—C17—C18	−177.6 (3)	C37—P2—C30—C35	−9.5 (3)
P1—C16—C21—C20	177.5 (3)	C37—P2—C44—C45	−104.4 (3)
P1—C23—C24—C25	−176.8 (3)	C37—P2—C44—C49	78.1 (3)
P1—C23—C28—C27	176.3 (3)	C37—C38—C39—C40	−1.2 (6)
P2—C30—C31—C32	178.4 (3)	C38—C37—C42—C41	2.9 (6)
P2—C30—C35—C34	−177.6 (3)	C38—C39—C40—C41	2.8 (6)
P2—C37—C38—C39	174.9 (3)	C38—C39—C40—C43	−175.8 (4)
P2—C37—C42—C41	−173.5 (3)	C39—C40—C41—C42	−1.6 (6)
P2—C44—C45—C46	−176.9 (3)	C40—C41—C42—C37	−1.3 (6)
P2—C44—C49—C48	177.2 (3)	C42—C37—C38—C39	−1.7 (6)
C9—P1—C16—C17	−23.4 (4)	C43—C40—C41—C42	177.1 (4)
C9—P1—C16—C21	160.3 (3)	C44—P2—C30—C31	63.7 (3)
C9—P1—C23—C24	54.1 (3)	C44—P2—C30—C35	−118.7 (3)
C9—P1—C23—C28	−123.0 (3)	C44—P2—C37—C38	−174.2 (3)
C9—C10—C11—C12	0.5 (5)	C44—P2—C37—C42	2.2 (4)
C10—C9—C14—C13	−1.0 (5)	C44—C45—C46—C47	0.1 (6)
C10—C11—C12—C13	−0.8 (5)	C45—C44—C49—C48	−0.4 (5)
C10—C11—C12—C15	−178.8 (3)	C45—C46—C47—C48	−1.2 (5)
C11—C12—C13—C14	0.1 (6)	C45—C46—C47—C50	177.9 (4)
C12—C13—C14—C9	0.8 (6)	C46—C47—C48—C49	1.4 (6)
C14—C9—C10—C11	0.4 (5)	C47—C48—C49—C44	−0.6 (6)
C15—C12—C13—C14	178.2 (4)	C49—C44—C45—C46	0.7 (5)
C16—P1—C9—C10	148.4 (3)	C50—C47—C48—C49	−177.7 (4)
C16—P1—C9—C14	−43.1 (3)	C51—C52—C53—C54	−0.9 (6)
C16—P1—C23—C24	−57.0 (3)	C52—C51—C56—C55	−0.9 (6)
C16—P1—C23—C28	125.8 (3)	C52—C53—C54—C55	0.3 (6)
C16—C17—C18—C19	0.4 (6)	C52—C53—C54—C58	178.2 (4)
C17—C16—C21—C20	1.0 (6)	C53—C54—C55—C56	−0.1 (6)
C17—C18—C19—C20	0.7 (6)	C54—C55—C56—C51	0.3 (6)
C17—C18—C19—C22	−177.6 (4)	C56—C51—C52—C53	1.2 (6)
C18—C19—C20—C21	−0.9 (6)	C57—C51—C52—C53	−178.5 (4)
C19—C20—C21—C16	0.1 (6)	C57—C51—C56—C55	178.8 (4)
C21—C16—C17—C18	−1.2 (6)	C58—C54—C55—C56	−177.9 (4)
C22—C19—C20—C21	177.3 (3)	C59—C60—C61—C59^{i}	−0.4 (8)
C23—P1—C9—C10	40.8 (3)	C59—C60—C61—C62	−179.2 (5)
C23—P1—C9—C14	−150.7 (3)	C61^{i}—C59—C60—C61	0.4 (8)
C23—P1—C16—C17	83.3 (3)		

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

Bis(*μ*-propanoato-1*κO*:2*κO′*)bis(tricarbonylosmium)(*Os*—*Os*) (5)

Crystal data

$[Os_2(C_3H_5O_2)_2(CO)_6]$
M_r = 694.60
Orthorhombic, *Pbca*
a = 9.79942 (10) Å
b = 15.51827 (19) Å
c = 21.7339 (2) Å
V = 3305.08 (6) Å^3
Z = 8
F(000) = 2512
D_x = 2.792 Mg m^{-3}
Cu *Kα* radiation, *λ* = 1.54184 Å
Cell parameters from 11012 reflections
θ = 5.7–73.4°
μ = 29.08 mm^{-1}
T = 100 K
Trapezoid, colorless
0.08 × 0.06 × 0.04 mm

Data collection

Rigaku SuperNova AtlasS2 CCD diffractometer
Radiation source: micro-focus sealed X-ray tube, SuperNova (Cu) X-ray Source
Mirror monochromator
Detector resolution: 5.2387 pixels mm^{-1}
ω scans
Absorption correction: multi-scan CrysAlisPro (Rigaku OD, 2015)
T_{min} = 0.637, T_{max} = 1.000
16571 measured reflections
3300 independent reflections
3196 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 73.7°, θ_{min} = 4.1°
h = −11→12
k = −18→19
l = −27→26

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.018
$wR(F^2)$ = 0.039
S = 1.03
3300 reflections
219 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0142P)^2 + 12.P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.93 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Os1	0.80792 (2)	0.55113 (2)	0.59651 (2)	0.00927 (5)
Os2	0.58526 (2)	0.65884 (2)	0.58989 (2)	0.00867 (5)
O1	0.7665 (3)	0.47459 (19)	0.46970 (12)	0.0240 (6)
O2	1.0011 (3)	0.68133 (19)	0.54088 (14)	0.0258 (6)
O3	1.0342 (3)	0.41963 (19)	0.63032 (15)	0.0298 (7)
O4	0.5287 (3)	0.60269 (19)	0.45938 (12)	0.0228 (6)
O5	0.7527 (3)	0.80956 (17)	0.54447 (13)	0.0204 (6)
O6	0.3175 (3)	0.76514 (18)	0.58797 (12)	0.0198 (6)
O7	0.4843 (2)	0.55431 (16)	0.63274 (12)	0.0141 (5)
O8	0.6683 (2)	0.46936 (16)	0.64223 (11)	0.0133 (5)

O9	0.6379 (2)	0.68838 (16)	0.68238 (11)	0.0139 (5)
O10	0.8253 (2)	0.60506 (16)	0.68545 (11)	0.0125 (5)
C1	0.7804 (3)	0.5025 (2)	0.51766 (17)	0.0163 (7)
C2	0.9285 (4)	0.6318 (2)	0.56154 (17)	0.0169 (7)
C3	0.9519 (4)	0.4673 (2)	0.61907 (17)	0.0167 (7)
C4	0.5482 (3)	0.6241 (2)	0.50883 (17)	0.0139 (7)
C5	0.6878 (3)	0.7526 (2)	0.56034 (16)	0.0136 (7)
C6	0.4139 (3)	0.7261 (2)	0.59352 (15)	0.0133 (7)
C7	0.5444 (3)	0.4876 (2)	0.65198 (15)	0.0128 (7)
C8	0.4621 (4)	0.4258 (2)	0.69069 (17)	0.0179 (7)
H8A	0.382532	0.407957	0.667429	0.021*
H8B	0.430190	0.455912	0.727057	0.021*
C9	0.5398 (5)	0.3462 (3)	0.7107 (2)	0.0339 (11)
H9A	0.563801	0.312751	0.675178	0.051*
H9B	0.483504	0.312306	0.737563	0.051*
H9C	0.621204	0.363151	0.732080	0.051*
C10	0.7394 (3)	0.6553 (2)	0.70948 (15)	0.0112 (6)
C11	0.7578 (3)	0.6738 (2)	0.77686 (16)	0.0141 (7)
H11A	0.726967	0.731813	0.786111	0.017*
H11B	0.853480	0.669456	0.787837	0.017*
C12	0.6742 (4)	0.6082 (3)	0.81360 (17)	0.0205 (8)
H12A	0.703193	0.550999	0.803112	0.031*
H12B	0.579226	0.614842	0.803915	0.031*
H12C	0.687896	0.617754	0.856809	0.031*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Os1	0.00868 (7)	0.00912 (8)	0.01001 (8)	0.00042 (5)	0.00130 (5)	0.00014 (5)
Os2	0.00900 (7)	0.00854 (8)	0.00847 (8)	0.00035 (5)	−0.00063 (5)	0.00031 (5)
O1	0.0343 (15)	0.0243 (15)	0.0134 (13)	0.0077 (12)	−0.0004 (12)	−0.0050 (11)
O2	0.0169 (13)	0.0243 (15)	0.0362 (17)	−0.0014 (11)	0.0072 (12)	0.0124 (13)
O3	0.0264 (15)	0.0216 (15)	0.0415 (18)	0.0127 (13)	−0.0082 (13)	0.0003 (13)
O4	0.0245 (13)	0.0281 (15)	0.0158 (13)	0.0045 (12)	−0.0081 (11)	−0.0098 (11)
O5	0.0189 (12)	0.0149 (13)	0.0275 (14)	−0.0041 (11)	0.0005 (11)	0.0054 (11)
O6	0.0147 (12)	0.0227 (14)	0.0219 (14)	0.0063 (11)	−0.0020 (10)	−0.0004 (11)
O7	0.0119 (11)	0.0116 (12)	0.0188 (12)	−0.0013 (9)	0.0019 (10)	0.0043 (10)
O8	0.0111 (11)	0.0115 (12)	0.0174 (12)	0.0000 (9)	0.0012 (9)	0.0033 (10)
O9	0.0153 (11)	0.0159 (12)	0.0104 (11)	0.0040 (10)	−0.0030 (9)	−0.0018 (9)
O10	0.0109 (11)	0.0163 (12)	0.0103 (11)	0.0018 (9)	−0.0020 (9)	−0.0021 (9)
C1	0.0144 (16)	0.0137 (17)	0.0208 (19)	0.0033 (13)	0.0034 (14)	0.0014 (15)
C2	0.0150 (17)	0.0150 (17)	0.0206 (18)	0.0061 (14)	−0.0001 (14)	0.0008 (15)
C3	0.0166 (17)	0.0160 (18)	0.0175 (17)	−0.0020 (15)	0.0024 (14)	−0.0001 (14)
C4	0.0096 (15)	0.0128 (16)	0.0195 (18)	0.0012 (13)	−0.0005 (13)	0.0017 (14)
C5	0.0146 (16)	0.0135 (17)	0.0128 (16)	0.0050 (14)	−0.0038 (13)	0.0003 (13)
C6	0.0147 (17)	0.0143 (17)	0.0109 (16)	−0.0035 (14)	0.0028 (13)	0.0004 (13)
C7	0.0138 (16)	0.0131 (16)	0.0116 (15)	−0.0049 (13)	−0.0012 (13)	−0.0024 (13)
C8	0.0165 (17)	0.0178 (18)	0.0193 (18)	−0.0037 (14)	0.0012 (14)	0.0058 (15)

C9	0.026 (2)	0.023 (2)	0.053 (3)	−0.0025 (17)	0.009 (2)	0.022 (2)
C10	0.0102 (14)	0.0118 (16)	0.0115 (15)	−0.0040 (12)	−0.0004 (13)	0.0019 (13)
C11	0.0132 (15)	0.0175 (17)	0.0115 (16)	0.0006 (14)	−0.0019 (13)	−0.0009 (14)
C12	0.0131 (16)	0.034 (2)	0.0141 (17)	−0.0032 (15)	0.0002 (14)	0.0042 (16)

Geometric parameters (Å, º)

Os1—Os2	2.7523 (2)	O8—C7	1.265 (4)
Os1—O8	2.114 (2)	O9—C10	1.265 (4)
Os1—O10	2.113 (2)	O10—C10	1.261 (4)
Os1—C1	1.891 (4)	C7—C8	1.509 (5)
Os1—C2	1.882 (4)	C8—H8A	0.9700
Os1—C3	1.981 (4)	C8—H8B	0.9700
Os2—O7	2.116 (2)	C8—C9	1.515 (5)
Os2—O9	2.125 (2)	C9—H9A	0.9600
Os2—C4	1.878 (4)	C9—H9B	0.9600
Os2—C5	1.882 (4)	C9—H9C	0.9600
Os2—C6	1.979 (4)	C10—C11	1.503 (5)
O1—C1	1.137 (5)	C11—H11A	0.9700
O2—C2	1.139 (5)	C11—H11B	0.9700
O3—C3	1.122 (5)	C11—C12	1.531 (5)
O4—C4	1.141 (5)	C12—H12A	0.9600
O5—C5	1.142 (5)	C12—H12B	0.9600
O6—C6	1.128 (4)	C12—H12C	0.9600
O7—C7	1.262 (4)		
O8—Os1—Os2	82.89 (6)	O2—C2—Os1	179.2 (4)
O10—Os1—Os2	82.61 (6)	O3—C3—Os1	178.3 (3)
O10—Os1—O8	81.95 (10)	O4—C4—Os2	178.5 (3)
C1—Os1—Os2	94.69 (10)	O5—C5—Os2	177.4 (3)
C1—Os1—O8	95.42 (13)	O6—C6—Os2	171.5 (3)
C1—Os1—O10	176.43 (12)	O7—C7—O8	125.2 (3)
C1—Os1—C3	93.66 (15)	O7—C7—C8	117.2 (3)
C2—Os1—Os2	94.16 (11)	O8—C7—C8	117.6 (3)
C2—Os1—O8	174.57 (13)	C7—C8—H8A	108.7
C2—Os1—O10	93.17 (13)	C7—C8—H8B	108.7
C2—Os1—C1	89.35 (16)	C7—C8—C9	114.2 (3)
C2—Os1—C3	95.16 (15)	H8A—C8—H8B	107.6
C3—Os1—Os2	167.54 (10)	C9—C8—H8A	108.7
C3—Os1—O8	87.14 (12)	C9—C8—H8B	108.7
C3—Os1—O10	88.64 (12)	C8—C9—H9A	109.5
O7—Os2—Os1	83.23 (7)	C8—C9—H9B	109.5
O7—Os2—O9	82.10 (10)	C8—C9—H9C	109.5
O9—Os2—Os1	83.63 (6)	H9A—C9—H9B	109.5
C4—Os2—Os1	91.60 (10)	H9A—C9—H9C	109.5
C4—Os2—O7	95.87 (13)	H9B—C9—H9C	109.5
C4—Os2—O9	174.99 (12)	O9—C10—C11	118.1 (3)
C4—Os2—C5	90.30 (15)	O10—C10—O9	125.7 (3)

C4—Os2—C6	91.42 (14)	O10—C10—C11	116.2 (3)
C5—Os2—Os1	93.68 (10)	C10—C11—H11A	110.0
C5—Os2—O7	173.16 (12)	C10—C11—H11B	110.0
C5—Os2—O9	91.51 (13)	C10—C11—C12	108.5 (3)
C5—Os2—C6	93.40 (14)	H11A—C11—H11B	108.4
C6—Os2—Os1	172.29 (10)	C12—C11—H11A	110.0
C6—Os2—O7	89.40 (12)	C12—C11—H11B	110.0
C6—Os2—O9	93.14 (12)	C11—C12—H12A	109.5
C7—O7—Os2	123.8 (2)	C11—C12—H12B	109.5
C7—O8—Os1	124.4 (2)	C11—C12—H12C	109.5
C10—O9—Os2	122.9 (2)	H12A—C12—H12B	109.5
C10—O10—Os1	124.7 (2)	H12A—C12—H12C	109.5
O1—C1—Os1	178.3 (3)	H12B—C12—H12C	109.5
Os1—O8—C7—O7	4.6 (5)	Os2—O9—C10—O10	3.7 (5)
Os1—O8—C7—C8	−174.2 (2)	Os2—O9—C10—C11	−173.6 (2)
Os1—O10—C10—O9	−7.9 (5)	O7—C7—C8—C9	178.5 (4)
Os1—O10—C10—C11	169.5 (2)	O8—C7—C8—C9	−2.7 (5)
Os2—O7—C7—O8	−8.7 (5)	O9—C10—C11—C12	86.1 (4)
Os2—O7—C7—C8	170.1 (2)	O10—C10—C11—C12	−91.5 (4)

Pentacarbonyl-1κ^2C,2κ^3C-bis(μ-propanoato-1κO:2$\kappa O'$)[tris(4-methylphenyl)phosphane-1κP]diosmium(*Os*—*Os*) (6)

Crystal data

$[Os_2(C_3H_5O_2)_2(C_{21}H_{21}P)(CO)_5]$
M_r = 970.94
Triclinic, $P\overline{1}$
a = 10.0952 (2) Å
b = 11.6630 (3) Å
c = 13.7536 (3) Å
α = 88.6185 (19)°
β = 86.7627 (18)°
γ = 86.9563 (19)°
V = 1614.12 (6) Å^3

Z = 2
$F(000)$ = 924
D_x = 1.998 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 22340 reflections
θ = 3.8–73.3°
μ = 15.55 mm^{-1}
T = 100 K
Block, colorless
0.21 × 0.14 × 0.06 mm

Data collection

Rigaku SuperNova AtlasS2 CCD diffractometer
Radiation source: micro-focus sealed X-ray tube, SuperNova (Cu) X-ray Source
Mirror monochromator
Detector resolution: 5.2387 pixels mm^{-1}
ω scans
Absorption correction: multi-scan CrysAlisPro (Rigaku OD, 2015)

T_{min} = 0.389, T_{max} = 1.000
30757 measured reflections
6461 independent reflections
6196 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 73.7°, θ_{min} = 3.8°
h = −12→12
k = −14→14
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.019

$wR(F^2)$ = 0.046
S = 1.08
6461 reflections

402 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0202P)^2 + 1.4592P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.55$ e Å^{-3}
$\Delta\rho_{min} = -1.21$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Os1	0.40363 (2)	0.24014 (2)	0.74050 (2)	0.01309 (4)
Os2	0.54412 (2)	0.18661 (2)	0.90234 (2)	0.01502 (4)
P1	0.24570 (6)	0.31901 (6)	0.62891 (5)	0.01422 (13)
O1	0.4095 (2)	0.00160 (17)	0.66128 (18)	0.0304 (5)
O2	0.6339 (2)	0.29582 (19)	0.60145 (16)	0.0265 (5)
O3	0.6587 (2)	−0.03401 (19)	0.81251 (16)	0.0290 (5)
O4	0.7726 (2)	0.31646 (19)	0.81165 (17)	0.0291 (5)
O5	0.6750 (2)	0.1397 (2)	1.09935 (16)	0.0305 (5)
O6	0.24276 (18)	0.20690 (17)	0.84390 (14)	0.0183 (4)
O7	0.36401 (19)	0.11713 (17)	0.95760 (15)	0.0208 (4)
O8	0.38423 (19)	0.40238 (16)	0.80787 (14)	0.0183 (4)
O9	0.44438 (18)	0.34012 (17)	0.95493 (15)	0.0194 (4)
C1	0.4078 (3)	0.0939 (3)	0.6913 (2)	0.0224 (6)
C2	0.5461 (3)	0.2749 (2)	0.6550 (2)	0.0198 (6)
C3	0.6153 (3)	0.0492 (3)	0.8479 (2)	0.0215 (6)
C4	0.6870 (3)	0.2654 (3)	0.8460 (2)	0.0213 (6)
C5	0.6204 (3)	0.1570 (2)	1.0308 (2)	0.0210 (6)
C6	0.2555 (3)	0.1515 (2)	0.9227 (2)	0.0204 (6)
C7	0.1308 (3)	0.1237 (4)	0.9828 (3)	0.0456 (11)
H7A	0.125794	0.040890	0.986624	0.055*
H7B	0.138925	0.150066	1.048443	0.055*
C8	0.0036 (3)	0.1740 (4)	0.9464 (3)	0.0415 (9)
H8A	−0.009125	0.144618	0.883182	0.062*
H8B	0.006905	0.256121	0.941912	0.062*
H8C	−0.068809	0.153792	0.990605	0.062*
C9	0.3874 (3)	0.4124 (2)	0.8986 (2)	0.0167 (5)
C10	0.3137 (3)	0.5149 (2)	0.9445 (2)	0.0222 (6)
H10A	0.303259	0.575955	0.895932	0.027*
H10B	0.364372	0.542942	0.995984	0.027*
C11	0.1783 (3)	0.4823 (3)	0.9860 (3)	0.0376 (8)
H11A	0.126660	0.458409	0.934307	0.056*
H11B	0.133755	0.547445	1.017205	0.056*
H11C	0.188798	0.420482	1.032689	0.056*
C12	0.1795 (3)	0.4602 (2)	0.6651 (2)	0.0174 (5)

C13	0.0499 (3)	0.4791 (3)	0.7035 (2)	0.0254 (6)
H13	−0.008069	0.419587	0.707137	0.030*
C14	0.0072 (3)	0.5875 (3)	0.7366 (3)	0.0337 (8)
H14	−0.079865	0.599664	0.761356	0.040*
C15	0.0914 (4)	0.6779 (3)	0.7334 (2)	0.0321 (7)
C16	0.2206 (3)	0.6582 (3)	0.6962 (2)	0.0267 (7)
H16	0.278265	0.717901	0.693574	0.032*
C17	0.2655 (3)	0.5516 (2)	0.6630 (2)	0.0211 (6)
H17	0.353074	0.539957	0.639044	0.025*
C18	0.0429 (5)	0.7949 (3)	0.7678 (3)	0.0512 (11)
H18A	0.069325	0.804868	0.832919	0.077*
H18B	−0.052191	0.801978	0.767068	0.077*
H18C	0.080683	0.852632	0.725414	0.077*
C19	0.1010 (3)	0.2370 (2)	0.6101 (2)	0.0176 (5)
C20	0.0678 (3)	0.1458 (3)	0.6720 (2)	0.0265 (7)
H20	0.118338	0.127549	0.725229	0.032*
C21	−0.0408 (3)	0.0815 (3)	0.6548 (2)	0.0285 (7)
H21	−0.062350	0.021569	0.697637	0.034*
C22	−0.1171 (3)	0.1047 (3)	0.5758 (2)	0.0218 (6)
C23	−0.0843 (3)	0.1965 (3)	0.5152 (2)	0.0278 (7)
H23	−0.135113	0.214581	0.462034	0.033*
C24	0.0224 (3)	0.2622 (3)	0.5318 (2)	0.0257 (7)
H24	0.041558	0.323730	0.490052	0.031*
C25	−0.2292 (3)	0.0303 (3)	0.5551 (2)	0.0292 (7)
H25A	−0.194827	−0.034993	0.518712	0.044*
H25B	−0.292855	0.073834	0.517934	0.044*
H25C	−0.271454	0.004677	0.615436	0.044*
C26	0.3104 (3)	0.3386 (2)	0.5033 (2)	0.0160 (5)
C27	0.3794 (3)	0.2460 (2)	0.4584 (2)	0.0191 (6)
H27	0.403098	0.181038	0.495291	0.023*
C28	0.4133 (3)	0.2495 (3)	0.3591 (2)	0.0223 (6)
H28	0.457846	0.186188	0.330034	0.027*
C29	0.3814 (3)	0.3467 (3)	0.3027 (2)	0.0211 (6)
C30	0.3178 (3)	0.4399 (2)	0.3482 (2)	0.0222 (6)
H30	0.299088	0.506516	0.311874	0.027*
C31	0.2810 (3)	0.4368 (2)	0.4474 (2)	0.0197 (6)
H31	0.236715	0.500351	0.476270	0.024*
C32	0.4146 (3)	0.3481 (3)	0.1945 (2)	0.0300 (7)
H32A	0.508795	0.335838	0.182509	0.045*
H32B	0.386097	0.421106	0.166994	0.045*
H32C	0.369938	0.288249	0.165069	0.045*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Os1	0.01340 (6)	0.01470 (6)	0.01134 (6)	−0.00004 (4)	−0.00365 (4)	0.00142 (4)
Os2	0.01319 (6)	0.01902 (7)	0.01305 (7)	0.00051 (4)	−0.00461 (4)	0.00201 (4)
P1	0.0142 (3)	0.0170 (3)	0.0116 (3)	−0.0003 (2)	−0.0032 (2)	0.0015 (2)

O1	0.0403 (13)	0.0159 (10)	0.0364 (13)	0.0034 (9)	−0.0165 (10)	−0.0060 (9)
O2	0.0228 (11)	0.0313 (12)	0.0249 (12)	−0.0051 (9)	0.0060 (9)	−0.0002 (9)
O3	0.0367 (12)	0.0260 (12)	0.0240 (12)	0.0076 (10)	−0.0060 (10)	−0.0028 (9)
O4	0.0209 (10)	0.0339 (12)	0.0329 (13)	−0.0064 (9)	−0.0046 (9)	0.0088 (10)
O5	0.0317 (12)	0.0401 (13)	0.0203 (12)	0.0020 (10)	−0.0131 (10)	0.0016 (9)
O6	0.0153 (9)	0.0249 (10)	0.0147 (10)	−0.0018 (8)	−0.0030 (7)	0.0033 (8)
O7	0.0173 (9)	0.0278 (11)	0.0174 (10)	−0.0010 (8)	−0.0033 (8)	0.0075 (8)
O8	0.0212 (10)	0.0171 (9)	0.0170 (10)	0.0010 (8)	−0.0044 (8)	−0.0016 (8)
O9	0.0179 (9)	0.0230 (10)	0.0175 (10)	0.0026 (8)	−0.0054 (8)	−0.0017 (8)
C1	0.0200 (14)	0.0298 (16)	0.0176 (14)	0.0006 (12)	−0.0085 (11)	0.0091 (12)
C2	0.0249 (14)	0.0157 (13)	0.0198 (14)	0.0002 (11)	−0.0090 (12)	−0.0030 (11)
C3	0.0201 (14)	0.0272 (16)	0.0179 (15)	−0.0034 (12)	−0.0083 (11)	0.0072 (12)
C4	0.0201 (14)	0.0249 (15)	0.0191 (15)	0.0041 (12)	−0.0075 (11)	0.0011 (11)
C5	0.0189 (13)	0.0242 (14)	0.0197 (15)	0.0028 (11)	−0.0030 (11)	0.0001 (11)
C6	0.0170 (13)	0.0247 (14)	0.0193 (15)	−0.0013 (11)	−0.0008 (11)	0.0026 (11)
C7	0.0237 (17)	0.067 (3)	0.044 (2)	0.0000 (17)	0.0035 (15)	0.031 (2)
C8	0.0178 (15)	0.064 (3)	0.042 (2)	−0.0049 (16)	0.0014 (14)	0.0141 (19)
C9	0.0127 (12)	0.0210 (13)	0.0168 (14)	−0.0021 (10)	−0.0025 (10)	−0.0025 (10)
C10	0.0202 (14)	0.0234 (14)	0.0232 (15)	0.0005 (11)	−0.0036 (11)	−0.0047 (12)
C11	0.0249 (16)	0.041 (2)	0.046 (2)	0.0017 (14)	0.0058 (15)	−0.0144 (17)
C12	0.0195 (13)	0.0205 (13)	0.0118 (13)	0.0054 (11)	−0.0043 (10)	0.0013 (10)
C13	0.0229 (15)	0.0332 (17)	0.0199 (15)	0.0019 (12)	−0.0016 (12)	−0.0039 (12)
C14	0.0277 (16)	0.046 (2)	0.0265 (17)	0.0140 (15)	−0.0017 (13)	−0.0100 (15)
C15	0.047 (2)	0.0279 (16)	0.0218 (16)	0.0122 (14)	−0.0142 (14)	−0.0063 (13)
C16	0.0395 (17)	0.0212 (14)	0.0204 (15)	0.0014 (13)	−0.0119 (13)	−0.0007 (12)
C17	0.0238 (14)	0.0208 (14)	0.0189 (15)	0.0027 (11)	−0.0063 (11)	0.0013 (11)
C18	0.064 (3)	0.041 (2)	0.047 (3)	0.025 (2)	−0.011 (2)	−0.0175 (18)
C19	0.0140 (12)	0.0222 (14)	0.0165 (14)	−0.0007 (10)	−0.0015 (10)	−0.0009 (11)
C20	0.0288 (16)	0.0280 (16)	0.0244 (16)	−0.0087 (13)	−0.0126 (13)	0.0075 (13)
C21	0.0304 (16)	0.0268 (16)	0.0298 (18)	−0.0102 (13)	−0.0101 (14)	0.0095 (13)
C22	0.0175 (13)	0.0257 (15)	0.0228 (15)	−0.0023 (11)	−0.0016 (11)	−0.0057 (12)
C23	0.0201 (14)	0.0410 (18)	0.0233 (16)	−0.0052 (13)	−0.0088 (12)	0.0057 (13)
C24	0.0211 (14)	0.0327 (16)	0.0237 (16)	−0.0060 (12)	−0.0065 (12)	0.0108 (13)
C25	0.0247 (15)	0.0340 (17)	0.0307 (18)	−0.0091 (13)	−0.0064 (13)	−0.0077 (14)
C26	0.0133 (12)	0.0215 (13)	0.0135 (13)	−0.0032 (10)	−0.0019 (10)	0.0009 (10)
C27	0.0208 (13)	0.0187 (13)	0.0181 (14)	−0.0001 (11)	−0.0047 (11)	0.0021 (11)
C28	0.0238 (14)	0.0239 (14)	0.0192 (15)	0.0010 (11)	−0.0013 (11)	−0.0028 (11)
C29	0.0205 (13)	0.0280 (15)	0.0154 (14)	−0.0060 (11)	−0.0024 (11)	0.0002 (11)
C30	0.0268 (15)	0.0211 (14)	0.0192 (15)	−0.0035 (12)	−0.0046 (12)	0.0059 (11)
C31	0.0226 (14)	0.0196 (13)	0.0169 (14)	0.0003 (11)	−0.0021 (11)	0.0014 (11)
C32	0.0383 (18)	0.0344 (17)	0.0171 (16)	−0.0037 (14)	0.0026 (13)	−0.0016 (13)

Geometric parameters (Å, º)

Os1—Os2	2.7479 (2)	C13—C14	1.392 (5)
Os1—P1	2.4075 (6)	C14—H14	0.9300
Os1—O6	2.1422 (19)	C14—C15	1.387 (5)
Os1—O8	2.1218 (19)	C15—C16	1.383 (5)

Os1—C1	1.848 (3)	C15—C18	1.503 (5)
Os1—C2	1.861 (3)	C16—H16	0.9300
Os2—O7	2.1239 (19)	C16—C17	1.380 (4)
Os2—O9	2.129 (2)	C17—H17	0.9300
Os2—C3	1.876 (3)	C18—H18A	0.9600
Os2—C4	1.872 (3)	C18—H18B	0.9600
Os2—C5	1.983 (3)	C18—H18C	0.9600
P1—C12	1.814 (3)	C19—C20	1.388 (4)
P1—C19	1.822 (3)	C19—C24	1.391 (4)
P1—C26	1.825 (3)	C20—H20	0.9300
O1—C1	1.161 (4)	C20—C21	1.395 (4)
O2—C2	1.152 (4)	C21—H21	0.9300
O3—C3	1.151 (4)	C21—C22	1.380 (4)
O4—C4	1.148 (4)	C22—C23	1.384 (4)
O5—C5	1.127 (4)	C22—C25	1.506 (4)
O6—C6	1.257 (3)	C23—H23	0.9300
O7—C6	1.263 (3)	C23—C24	1.387 (4)
O8—C9	1.259 (3)	C24—H24	0.9300
O9—C9	1.268 (3)	C25—H25A	0.9600
C6—C7	1.512 (4)	C25—H25B	0.9600
C7—H7A	0.9700	C25—H25C	0.9600
C7—H7B	0.9700	C26—C27	1.392 (4)
C7—C8	1.491 (5)	C26—C31	1.392 (4)
C8—H8A	0.9600	C27—H27	0.9300
C8—H8B	0.9600	C27—C28	1.389 (4)
C8—H8C	0.9600	C28—H28	0.9300
C9—C10	1.507 (4)	C28—C29	1.393 (4)
C10—H10A	0.9700	C29—C30	1.379 (4)
C10—H10B	0.9700	C29—C32	1.506 (4)
C10—C11	1.515 (4)	C30—H30	0.9300
C11—H11A	0.9600	C30—C31	1.394 (4)
C11—H11B	0.9600	C31—H31	0.9300
C11—H11C	0.9600	C32—H32A	0.9600
C12—C13	1.392 (4)	C32—H32B	0.9600
C12—C17	1.409 (4)	C32—H32C	0.9600
C13—H13	0.9300		

P1—Os1—Os2	164.673 (18)	H11B—C11—H11C	109.5
O6—Os1—Os2	80.26 (5)	C13—C12—P1	122.2 (2)
O6—Os1—P1	89.31 (5)	C13—C12—C17	118.6 (3)
O8—Os1—Os2	81.23 (5)	C17—C12—P1	118.9 (2)
O8—Os1—P1	86.12 (5)	C12—C13—H13	120.1
O8—Os1—O6	81.16 (8)	C12—C13—C14	119.7 (3)
C1—Os1—Os2	97.27 (8)	C14—C13—H13	120.1
C1—Os1—P1	94.45 (8)	C13—C14—H14	119.2
C1—Os1—O6	93.03 (11)	C15—C14—C13	121.7 (3)
C1—Os1—O8	174.16 (11)	C15—C14—H14	119.2
C1—Os1—C2	89.68 (13)	C14—C15—C18	120.9 (4)

C2—Os1—Os2	98.10 (8)	C16—C15—C14	118.3 (3)
C2—Os1—P1	91.79 (8)	C16—C15—C18	120.8 (3)
C2—Os1—O6	176.99 (10)	C15—C16—H16	119.4
C2—Os1—O8	96.11 (10)	C17—C16—C15	121.3 (3)
O7—Os2—Os1	84.05 (5)	C17—C16—H16	119.4
O7—Os2—O9	81.07 (8)	C12—C17—H17	119.8
O9—Os2—Os1	82.62 (5)	C16—C17—C12	120.4 (3)
C3—Os2—Os1	91.82 (8)	C16—C17—H17	119.8
C3—Os2—O7	95.21 (10)	C15—C18—H18A	109.5
C3—Os2—O9	173.58 (9)	C15—C18—H18B	109.5
C3—Os2—C5	94.90 (12)	C15—C18—H18C	109.5
C4—Os2—Os1	89.46 (8)	H18A—C18—H18B	109.5
C4—Os2—O7	171.57 (10)	H18A—C18—H18C	109.5
C4—Os2—O9	92.79 (11)	H18B—C18—H18C	109.5
C4—Os2—C3	90.36 (13)	C20—C19—P1	121.1 (2)
C4—Os2—C5	96.32 (12)	C20—C19—C24	118.0 (2)
C5—Os2—Os1	171.10 (9)	C24—C19—P1	120.8 (2)
C5—Os2—O7	89.52 (10)	C19—C20—H20	119.8
C5—Os2—O9	90.31 (10)	C19—C20—C21	120.4 (3)
C12—P1—Os1	111.59 (9)	C21—C20—H20	119.8
C12—P1—C19	105.33 (13)	C20—C21—H21	119.2
C12—P1—C26	104.93 (13)	C22—C21—C20	121.6 (3)
C19—P1—Os1	117.64 (9)	C22—C21—H21	119.2
C19—P1—C26	100.50 (12)	C21—C22—C23	117.6 (3)
C26—P1—Os1	115.42 (9)	C21—C22—C25	121.0 (3)
C6—O6—Os1	124.22 (18)	C23—C22—C25	121.5 (3)
C6—O7—Os2	119.70 (18)	C22—C23—H23	119.2
C9—O8—Os1	122.11 (18)	C22—C23—C24	121.6 (3)
C9—O9—Os2	121.98 (19)	C24—C23—H23	119.2
O1—C1—Os1	179.2 (3)	C19—C24—H24	119.6
O2—C2—Os1	179.4 (3)	C23—C24—C19	120.7 (3)
O3—C3—Os2	178.5 (2)	C23—C24—H24	119.6
O4—C4—Os2	178.1 (3)	C22—C25—H25A	109.5
O5—C5—Os2	173.6 (3)	C22—C25—H25B	109.5
O6—C6—O7	125.9 (3)	C22—C25—H25C	109.5
O6—C6—C7	118.0 (3)	H25A—C25—H25B	109.5
O7—C6—C7	116.0 (3)	H25A—C25—H25C	109.5
C6—C7—H7A	108.3	H25B—C25—H25C	109.5
C6—C7—H7B	108.3	C27—C26—P1	118.1 (2)
H7A—C7—H7B	107.4	C31—C26—P1	122.9 (2)
C8—C7—C6	115.9 (3)	C31—C26—C27	118.6 (3)
C8—C7—H7A	108.3	C26—C27—H27	119.6
C8—C7—H7B	108.3	C28—C27—C26	120.8 (3)
C7—C8—H8A	109.5	C28—C27—H27	119.6
C7—C8—H8B	109.5	C27—C28—H28	119.7
C7—C8—H8C	109.5	C27—C28—C29	120.7 (3)
H8A—C8—H8B	109.5	C29—C28—H28	119.7
H8A—C8—H8C	109.5	C28—C29—C32	120.3 (3)

H8B—C8—H8C	109.5	C30—C29—C28	118.3 (3)
O8—C9—O9	124.7 (3)	C30—C29—C32	121.4 (3)
O8—C9—C10	117.8 (2)	C29—C30—H30	119.2
O9—C9—C10	117.4 (3)	C29—C30—C31	121.6 (3)
C9—C10—H10A	109.6	C31—C30—H30	119.2
C9—C10—H10B	109.6	C26—C31—C30	120.0 (3)
C9—C10—C11	110.2 (2)	C26—C31—H31	120.0
H10A—C10—H10B	108.1	C30—C31—H31	120.0
C11—C10—H10A	109.6	C29—C32—H32A	109.5
C11—C10—H10B	109.6	C29—C32—H32B	109.5
C10—C11—H11A	109.5	C29—C32—H32C	109.5
C10—C11—H11B	109.5	H32A—C32—H32B	109.5
C10—C11—H11C	109.5	H32A—C32—H32C	109.5
H11A—C11—H11B	109.5	H32B—C32—H32C	109.5
H11A—C11—H11C	109.5		

Os1—P1—C12—C13	−107.8 (2)	C13—C14—C15—C16	0.0 (5)
Os1—P1—C12—C17	66.0 (2)	C13—C14—C15—C18	178.9 (3)
Os1—P1—C19—C20	13.3 (3)	C14—C15—C16—C17	0.0 (5)
Os1—P1—C19—C24	−165.1 (2)	C15—C16—C17—C12	0.8 (4)
Os1—P1—C26—C27	50.8 (2)	C17—C12—C13—C14	1.5 (4)
Os1—P1—C26—C31	−136.7 (2)	C18—C15—C16—C17	−178.9 (3)
Os1—O6—C6—O7	7.4 (4)	C19—P1—C12—C13	20.9 (3)
Os1—O6—C6—C7	−173.3 (3)	C19—P1—C12—C17	−165.2 (2)
Os1—O8—C9—O9	25.0 (3)	C19—P1—C26—C27	−76.8 (2)
Os1—O8—C9—C10	−152.37 (18)	C19—P1—C26—C31	95.7 (2)
Os2—O7—C6—O6	15.6 (4)	C19—C20—C21—C22	1.0 (6)
Os2—O7—C6—C7	−163.7 (3)	C20—C19—C24—C23	−1.2 (5)
Os2—O9—C9—O8	−0.6 (4)	C20—C21—C22—C23	−1.8 (5)
Os2—O9—C9—C10	176.83 (17)	C20—C21—C22—C25	176.5 (3)
P1—C12—C13—C14	175.4 (2)	C21—C22—C23—C24	1.0 (5)
P1—C12—C17—C16	−175.6 (2)	C22—C23—C24—C19	0.5 (5)
P1—C19—C20—C21	−177.9 (3)	C24—C19—C20—C21	0.5 (5)
P1—C19—C24—C23	177.2 (3)	C25—C22—C23—C24	−177.3 (3)
P1—C26—C27—C28	170.1 (2)	C26—P1—C12—C13	126.5 (2)
P1—C26—C31—C30	−171.0 (2)	C26—P1—C12—C17	−59.7 (2)
O6—C6—C7—C8	−5.1 (5)	C26—P1—C19—C20	139.4 (3)
O7—C6—C7—C8	174.3 (3)	C26—P1—C19—C24	−38.9 (3)
O8—C9—C10—C11	97.4 (3)	C26—C27—C28—C29	1.3 (4)
O9—C9—C10—C11	−80.2 (3)	C27—C26—C31—C30	1.5 (4)
C12—P1—C19—C20	−111.7 (3)	C27—C28—C29—C30	1.3 (4)
C12—P1—C19—C24	69.9 (3)	C27—C28—C29—C32	−178.0 (3)
C12—P1—C26—C27	174.1 (2)	C28—C29—C30—C31	−2.6 (4)
C12—P1—C26—C31	−13.4 (2)	C29—C30—C31—C26	1.2 (4)
C12—C13—C14—C15	−0.8 (5)	C31—C26—C27—C28	−2.7 (4)
C13—C12—C17—C16	−1.6 (4)	C32—C29—C30—C31	176.7 (3)

Bis(*μ*-propanoato-1*κO*:2*κO′*)bis[tris(4-methylphenyl)phosphane]-1*κP*,2*κP*-bis(dicarbonylosmium)(*Os—Os*), dichloromethane monosolvate (7@CH2Cl2)

Crystal data

$[Os_2(C_3H_5O_2)_2(C_{21}H_{21}P)_2(CO)_4]\cdot CH_2Cl_2$
M_r = 1332.20
Monoclinic, $P2_1/c$
a = 18.4968 (6) Å
b = 18.7369 (7) Å
c = 14.9214 (6) Å
β = 91.806 (2)°
V = 5168.8 (3) Å^3
Z = 4
$F(000)$ = 2608
D_x = 1.712 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 11533 reflections
θ = 1.6–27.6°
μ = 5.13 mm^{-1}
T = 100 K
Prisms, colorless
0.28 × 0.12 × 0.09 mm

Data collection

Rigaku SCX-Mini Mercury 2+ CCD diffractometer
Radiation source: sealed tube
ω–scans
Absorption correction: multi-scan (ABSCOR; Higashi, 2001)
T_{min} = 0.673, T_{max} = 1.00
54214 measured reflections
11794 independent reflections
9639 reflections with $I > 2\sigma(I)$
R_{int} = 0.060
θ_{max} = 27.5°, θ_{min} = 3.1°
h = −24→24
k = −23→24
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.071
S = 1.07
11794 reflections
624 parameters
20 restraints
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0224P)^2 + 9.8357P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 1.39 e Å^{-3}
$\Delta\rho_{min}$ = −1.32 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.7088 (2)	0.4345 (2)	−0.0552 (3)	0.0150 (9)	
C2	0.6426 (2)	0.4242 (2)	−0.0150 (3)	0.0218 (10)	
H2	0.6372	0.4380	0.0457	0.026*	
C3	0.5843 (3)	0.3938 (3)	−0.0628 (3)	0.0248 (11)	
H3	0.5397	0.3874	−0.0340	0.030*	
C4	0.5903 (2)	0.3728 (2)	−0.1516 (3)	0.0202 (10)	
C5	0.6558 (3)	0.3852 (3)	−0.1917 (3)	0.0247 (11)	
H5	0.6608	0.3727	−0.2529	0.030*	
C6	0.7143 (3)	0.4154 (2)	−0.1445 (3)	0.0216 (10)	

H6	0.7585	0.4231	−0.1738	0.026*
C7	0.5284 (2)	0.3369 (3)	−0.2015 (3)	0.0236 (11)
H7A	0.5338	0.2850	−0.1967	0.035*
H7B	0.5285	0.3509	−0.2648	0.035*
H7C	0.4826	0.3513	−0.1756	0.035*
C8	0.8199 (2)	0.5373 (2)	−0.0699 (3)	0.0153 (9)
C9	0.8615 (2)	0.5187 (2)	−0.1427 (3)	0.0180 (9)
H9	0.8780	0.4710	−0.1488	0.022*
C10	0.8790 (2)	0.5699 (2)	−0.2067 (3)	0.0190 (10)
H10	0.9057	0.5557	−0.2571	0.023*
C11	0.8585 (2)	0.6407 (2)	−0.1988 (3)	0.0188 (10)
C12	0.8190 (2)	0.6591 (2)	−0.1246 (3)	0.0176 (10)
H12	0.8053	0.7074	−0.1165	0.021*
C13	0.7991 (2)	0.6088 (2)	−0.0624 (3)	0.0160 (9)
H13	0.7707	0.6230	−0.0135	0.019*
C14	0.8760 (3)	0.6936 (3)	−0.2702 (3)	0.0281 (12)
H14A	0.9130	0.6737	−0.3085	0.042*
H14B	0.8941	0.7377	−0.2422	0.042*
H14C	0.8322	0.7041	−0.3066	0.042*
C15	0.8541 (2)	0.4003 (2)	0.0034 (3)	0.0159 (9)
C16	0.8354 (3)	0.3286 (2)	−0.0059 (3)	0.0197 (10)
H16	0.7860	0.3157	−0.0142	0.024*
C17	0.8879 (3)	0.2756 (2)	−0.0031 (3)	0.0240 (11)
H17	0.8738	0.2271	−0.0101	0.029*
C18	0.9605 (3)	0.2921 (2)	0.0097 (3)	0.0216 (10)
C19	0.9795 (3)	0.3640 (3)	0.0204 (3)	0.0219 (10)
H19	1.0290	0.3767	0.0288	0.026*
C20	0.9271 (2)	0.4168 (2)	0.0189 (3)	0.0201 (10)
H20	0.9410	0.4651	0.0286	0.024*
C21	1.0178 (3)	0.2344 (3)	0.0113 (4)	0.0348 (13)
H21A	1.0485	0.2400	−0.0406	0.052*
H21B	0.9945	0.1875	0.0090	0.052*
H21C	1.0476	0.2384	0.0666	0.052*
C22	0.8191 (2)	0.4573 (2)	0.5615 (3)	0.0167 (9)
C23	0.8132 (2)	0.4047 (2)	0.6263 (3)	0.0206 (10)
H23	0.7676	0.3829	0.6351	0.025*
C24	0.8732 (2)	0.3834 (2)	0.6785 (3)	0.0213 (10)
H24	0.8681	0.3469	0.7220	0.026*
C25	0.9404 (2)	0.4152 (2)	0.6676 (3)	0.0224 (10)
C26	0.9467 (3)	0.4665 (2)	0.6006 (3)	0.0237 (11)
H26	0.9925	0.4874	0.5904	0.028*
C27	0.8867 (2)	0.4875 (2)	0.5484 (3)	0.0200 (10)
H27	0.8920	0.5228	0.5034	0.024*
C28	1.0047 (3)	0.3951 (3)	0.7271 (3)	0.0312 (12)
H28A	0.9886	0.3856	0.7879	0.047*
H28B	1.0396	0.4345	0.7288	0.047*
H28C	1.0277	0.3523	0.7034	0.047*
C29	0.7175 (2)	0.5702 (2)	0.5717 (3)	0.0166 (9)

C30	0.7101 (2)	0.5582 (3)	0.6632 (3)	0.0205 (10)	
H30	0.7193	0.5119	0.6870	0.025*	
C31	0.6898 (2)	0.6121 (2)	0.7198 (3)	0.0216 (10)	
H31	0.6835	0.6021	0.7815	0.026*	
C32	0.6784 (3)	0.6816 (3)	0.6875 (3)	0.0242 (11)	
C33	0.6863 (3)	0.6937 (2)	0.5969 (3)	0.0247 (11)	
H33	0.6786	0.7403	0.5735	0.030*	
C34	0.7056 (3)	0.6388 (2)	0.5392 (3)	0.0218 (10)	
H34	0.7105	0.6483	0.4771	0.026*	
C35	0.6576 (3)	0.7410 (3)	0.7489 (4)	0.0358 (14)	
H35A	0.6086	0.7327	0.7698	0.054*	
H35B	0.6589	0.7865	0.7166	0.054*	
H35C	0.6916	0.7427	0.8005	0.054*	
C36	0.6674 (2)	0.4326 (2)	0.5165 (3)	0.0182 (10)	
C37	0.6754 (3)	0.3610 (2)	0.4919 (3)	0.0239 (11)	
H37	0.7210	0.3440	0.4733	0.029*	
C38	0.6167 (3)	0.3149 (3)	0.4947 (3)	0.0277 (12)	
H38	0.6222	0.2669	0.4757	0.033*	
C39	0.5499 (3)	0.3375 (3)	0.5249 (3)	0.0253 (11)	
C40	0.5429 (3)	0.4081 (3)	0.5487 (3)	0.0235 (11)	
H40	0.4977	0.4247	0.5689	0.028*	
C41	0.6002 (2)	0.4555 (3)	0.5438 (3)	0.0216 (10)	
H41	0.5934	0.5042	0.5592	0.026*	
C42	0.4871 (3)	0.2861 (3)	0.5288 (4)	0.0396 (14)	
H42A	0.4449	0.3108	0.5520	0.059*	
H42B	0.5002	0.2461	0.5683	0.059*	
H42C	0.4753	0.2680	0.4684	0.059*	
C43	0.7013 (2)	0.3863 (2)	0.2485 (3)	0.0200 (10)	
C44	0.6742 (3)	0.3105 (3)	0.2501 (4)	0.0333 (13)	
H44A	0.7102	0.2812	0.2796	0.040*	0.73
H44B	0.6317	0.3090	0.2855	0.040*	0.73
H44C	0.6244	0.3098	0.2298	0.040*	0.27
H44D	0.6764	0.2933	0.3108	0.040*	0.27
C45	0.6568 (6)	0.2799 (4)	0.1632 (5)	0.050 (2)	0.73
H45A	0.6211	0.3101	0.1314	0.074*	0.73
H45B	0.6368	0.2320	0.1708	0.074*	0.73
H45C	0.7007	0.2770	0.1284	0.074*	0.73
C45A	0.7161 (12)	0.2623 (7)	0.1944 (17)	0.053 (6)	0.27
H45D	0.7320	0.2880	0.1414	0.079*	0.27
H45E	0.6859	0.2217	0.1756	0.079*	0.27
H45F	0.7585	0.2450	0.2291	0.079*	0.27
C46	0.8792 (2)	0.4331 (2)	0.2770 (3)	0.0152 (9)	
C47	0.9384 (2)	0.3784 (2)	0.2877 (3)	0.0211 (10)	
H47A	0.9586	0.3797	0.3499	0.025*	
H47B	0.9778	0.3897	0.2467	0.025*	
C48	0.9092 (3)	0.3045 (3)	0.2671 (4)	0.0360 (13)	
H48A	0.8704	0.2933	0.3080	0.054*	
H48B	0.9482	0.2694	0.2748	0.054*	

H48C	0.8901	0.3031	0.2051	0.054*
C49	0.8253 (3)	0.5936 (3)	0.1509 (3)	0.0203 (10)
C50	0.6878 (3)	0.5657 (2)	0.1278 (3)	0.0195 (10)
C51	0.8005 (3)	0.6073 (2)	0.3551 (3)	0.0194 (10)
C52	0.6668 (3)	0.5639 (2)	0.3303 (3)	0.0177 (10)
C53	0.5166 (3)	0.9665 (3)	0.2748 (4)	0.0437 (15)
H53A	0.4810	1.0036	0.2903	0.052*
H53B	0.5619	0.9909	0.2588	0.052*
O1	0.71078 (19)	0.41452 (16)	0.1737 (2)	0.0250 (8)
O2	0.71458 (19)	0.41517 (17)	0.3238 (2)	0.0257 (8)
O3	0.85738 (17)	0.44616 (16)	0.1974 (2)	0.0215 (7)
O4	0.85480 (16)	0.46159 (16)	0.3465 (2)	0.0199 (7)
O5	0.86026 (19)	0.64365 (18)	0.1453 (2)	0.0301 (8)
O6	0.63722 (19)	0.5994 (2)	0.1074 (2)	0.0362 (9)
O7	0.83055 (18)	0.66144 (17)	0.3629 (2)	0.0278 (8)
O8	0.61028 (17)	0.59124 (18)	0.3218 (2)	0.0266 (8)
Os1	0.76852 (2)	0.51169 (2)	0.15852 (2)	0.01284 (5)
Os2	0.75547 (2)	0.51941 (2)	0.34254 (2)	0.01333 (5)
P1	0.78714 (6)	0.47126 (6)	0.00812 (8)	0.0143 (2)
P2	0.74144 (6)	0.49529 (6)	0.49914 (8)	0.0156 (2)
Cl1	0.48329 (8)	0.91824 (9)	0.18186 (11)	0.0510 (4)
Cl2	0.53392 (11)	0.91213 (11)	0.36866 (11)	0.0674 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.016 (2)	0.012 (2)	0.016 (2)	0.0012 (16)	−0.0008 (18)	−0.0003 (17)
C2	0.019 (3)	0.029 (3)	0.017 (2)	0.0015 (19)	0.0021 (19)	−0.003 (2)
C3	0.018 (3)	0.032 (3)	0.025 (3)	−0.002 (2)	0.004 (2)	−0.003 (2)
C4	0.018 (3)	0.019 (2)	0.023 (3)	−0.0001 (18)	−0.004 (2)	0.001 (2)
C5	0.024 (3)	0.034 (3)	0.016 (2)	−0.001 (2)	−0.002 (2)	−0.005 (2)
C6	0.023 (3)	0.026 (3)	0.016 (2)	−0.0069 (19)	0.003 (2)	−0.002 (2)
C7	0.016 (3)	0.032 (3)	0.023 (3)	−0.001 (2)	0.000 (2)	−0.006 (2)
C8	0.017 (2)	0.017 (2)	0.012 (2)	−0.0020 (17)	−0.0046 (17)	−0.0024 (18)
C9	0.016 (2)	0.017 (2)	0.021 (2)	−0.0023 (18)	0.0004 (18)	0.0006 (19)
C10	0.022 (3)	0.023 (2)	0.012 (2)	−0.0041 (19)	0.0058 (19)	−0.0026 (19)
C11	0.014 (2)	0.022 (2)	0.020 (2)	−0.0040 (18)	−0.0023 (19)	0.007 (2)
C12	0.015 (2)	0.018 (2)	0.020 (2)	0.0005 (17)	−0.0042 (19)	−0.0013 (19)
C13	0.016 (2)	0.021 (2)	0.011 (2)	−0.0004 (18)	0.0025 (18)	−0.0006 (18)
C14	0.030 (3)	0.027 (3)	0.028 (3)	0.003 (2)	0.006 (2)	0.011 (2)
C15	0.020 (2)	0.020 (2)	0.008 (2)	−0.0017 (18)	0.0016 (18)	0.0016 (18)
C16	0.022 (3)	0.023 (2)	0.014 (2)	−0.0025 (19)	0.0003 (19)	−0.0002 (19)
C17	0.033 (3)	0.017 (2)	0.022 (3)	0.002 (2)	0.000 (2)	−0.001 (2)
C18	0.026 (3)	0.025 (3)	0.014 (2)	0.009 (2)	0.000 (2)	0.000 (2)
C19	0.017 (3)	0.033 (3)	0.015 (2)	0.002 (2)	0.0001 (19)	−0.001 (2)
C20	0.019 (3)	0.023 (2)	0.019 (2)	−0.0007 (19)	0.0002 (19)	−0.001 (2)
C21	0.035 (3)	0.033 (3)	0.036 (3)	0.015 (2)	0.001 (3)	−0.003 (3)
C22	0.020 (2)	0.016 (2)	0.014 (2)	0.0049 (18)	0.0011 (18)	0.0007 (18)

C23	0.016 (2)	0.021 (2)	0.026 (3)	0.0002 (18)	0.003 (2)	0.001 (2)
C24	0.021 (3)	0.022 (2)	0.021 (3)	0.0046 (19)	0.002 (2)	0.002 (2)
C25	0.019 (3)	0.025 (3)	0.022 (3)	0.0061 (19)	−0.001 (2)	−0.006 (2)
C26	0.020 (3)	0.028 (3)	0.024 (3)	0.0023 (19)	0.006 (2)	−0.006 (2)
C27	0.023 (3)	0.024 (2)	0.013 (2)	0.0016 (19)	0.0044 (19)	−0.0013 (19)
C28	0.030 (3)	0.035 (3)	0.028 (3)	0.005 (2)	−0.006 (2)	0.003 (2)
C29	0.014 (2)	0.021 (2)	0.015 (2)	0.0025 (17)	0.0036 (18)	−0.0048 (19)
C30	0.016 (2)	0.028 (3)	0.018 (2)	0.0056 (19)	0.0017 (19)	0.001 (2)
C31	0.022 (3)	0.031 (3)	0.013 (2)	0.004 (2)	0.0011 (19)	−0.005 (2)
C32	0.017 (3)	0.032 (3)	0.023 (3)	0.007 (2)	−0.007 (2)	−0.006 (2)
C33	0.031 (3)	0.018 (2)	0.025 (3)	0.002 (2)	−0.002 (2)	−0.003 (2)
C34	0.025 (3)	0.021 (2)	0.019 (3)	0.0049 (19)	0.001 (2)	0.001 (2)
C35	0.041 (3)	0.038 (3)	0.028 (3)	0.016 (3)	−0.004 (3)	−0.013 (2)
C36	0.022 (3)	0.019 (2)	0.014 (2)	0.0020 (18)	0.0030 (19)	0.0047 (19)
C37	0.024 (3)	0.022 (2)	0.027 (3)	0.0011 (19)	0.010 (2)	0.005 (2)
C38	0.030 (3)	0.021 (2)	0.032 (3)	−0.004 (2)	0.008 (2)	0.000 (2)
C39	0.023 (3)	0.027 (3)	0.026 (3)	−0.005 (2)	0.000 (2)	0.005 (2)
C40	0.016 (3)	0.035 (3)	0.020 (3)	0.001 (2)	0.003 (2)	−0.001 (2)
C41	0.023 (3)	0.021 (2)	0.021 (3)	0.0030 (19)	0.000 (2)	0.005 (2)
C42	0.032 (3)	0.037 (3)	0.051 (4)	−0.011 (2)	0.010 (3)	−0.001 (3)
C43	0.018 (3)	0.014 (2)	0.028 (3)	−0.0045 (18)	0.008 (2)	0.001 (2)
C44	0.043 (3)	0.020 (3)	0.037 (3)	−0.010 (2)	0.011 (3)	−0.005 (2)
C45	0.092 (7)	0.030 (4)	0.027 (4)	−0.026 (4)	−0.007 (4)	0.006 (3)
C45A	0.066 (12)	0.029 (9)	0.064 (12)	−0.009 (9)	0.018 (10)	−0.005 (9)
C46	0.017 (2)	0.012 (2)	0.017 (2)	−0.0012 (16)	0.0025 (18)	−0.0002 (18)
C47	0.020 (3)	0.025 (3)	0.019 (2)	0.0062 (19)	−0.001 (2)	0.001 (2)
C48	0.042 (3)	0.023 (3)	0.042 (3)	0.001 (2)	−0.010 (3)	0.001 (2)
C49	0.021 (3)	0.028 (3)	0.012 (2)	−0.007 (2)	0.0004 (19)	−0.002 (2)
C50	0.021 (3)	0.028 (3)	0.010 (2)	−0.004 (2)	0.0054 (19)	−0.0076 (19)
C51	0.027 (3)	0.022 (2)	0.009 (2)	−0.001 (2)	0.0046 (19)	−0.0026 (19)
C52	0.028 (3)	0.017 (2)	0.009 (2)	−0.0085 (19)	0.0048 (19)	−0.0014 (18)
C53	0.053 (4)	0.040 (3)	0.038 (4)	−0.001 (3)	−0.003 (3)	−0.006 (3)
O1	0.041 (2)	0.0179 (17)	0.0159 (17)	−0.0130 (15)	0.0039 (15)	−0.0058 (14)
O2	0.037 (2)	0.0210 (17)	0.0198 (18)	−0.0073 (15)	0.0066 (15)	0.0020 (14)
O3	0.0251 (18)	0.0230 (17)	0.0166 (17)	0.0116 (14)	0.0019 (14)	−0.0008 (14)
O4	0.0206 (17)	0.0217 (17)	0.0174 (17)	0.0080 (13)	0.0015 (13)	0.0000 (14)
O5	0.041 (2)	0.0255 (19)	0.024 (2)	−0.0136 (16)	−0.0038 (17)	−0.0009 (15)
O6	0.027 (2)	0.055 (2)	0.027 (2)	0.0235 (18)	−0.0029 (16)	−0.0073 (18)
O7	0.030 (2)	0.0217 (18)	0.032 (2)	−0.0101 (15)	0.0047 (16)	−0.0036 (15)
O8	0.0180 (19)	0.036 (2)	0.0257 (19)	0.0077 (15)	0.0031 (15)	0.0073 (16)
Os1	0.01399 (9)	0.01247 (9)	0.01209 (9)	−0.00015 (6)	0.00080 (6)	−0.00065 (7)
Os2	0.01522 (10)	0.01266 (9)	0.01222 (9)	0.00003 (6)	0.00198 (7)	−0.00003 (7)
P1	0.0148 (6)	0.0143 (6)	0.0138 (6)	−0.0006 (4)	0.0017 (4)	−0.0020 (4)
P2	0.0163 (6)	0.0161 (6)	0.0145 (6)	0.0015 (4)	0.0026 (5)	0.0003 (5)
Cl1	0.0441 (9)	0.0578 (10)	0.0507 (10)	−0.0114 (7)	−0.0054 (8)	−0.0148 (8)
Cl2	0.0841 (14)	0.0830 (13)	0.0365 (9)	0.0053 (10)	0.0230 (9)	0.0165 (9)

Geometric parameters (Å, º)

C1—C6	1.387 (6)	C32—C33	1.383 (7)
C1—C2	1.393 (6)	C32—C35	1.500 (6)
C1—P1	1.838 (4)	C33—C34	1.395 (6)
C2—C3	1.395 (6)	C33—H33	0.9500
C2—H2	0.9500	C34—H34	0.9500
C3—C4	1.390 (7)	C35—H35A	0.9800
C3—H3	0.9500	C35—H35B	0.9800
C4—C5	1.388 (7)	C35—H35C	0.9800
C4—C7	1.505 (6)	C36—C41	1.388 (6)
C5—C6	1.394 (6)	C36—C37	1.400 (6)
C5—H5	0.9500	C36—P2	1.829 (5)
C6—H6	0.9500	C37—C38	1.390 (6)
C7—H7A	0.9800	C37—H37	0.9500
C7—H7B	0.9800	C38—C39	1.394 (7)
C7—H7C	0.9800	C38—H38	0.9500
C8—C9	1.394 (6)	C39—C40	1.376 (7)
C8—C13	1.399 (6)	C39—C42	1.512 (7)
C8—P1	1.816 (4)	C40—C41	1.388 (6)
C9—C10	1.400 (6)	C40—H40	0.9500
C9—H9	0.9500	C41—H41	0.9500
C10—C11	1.386 (6)	C42—H42A	0.9800
C10—H10	0.9500	C42—H42B	0.9800
C11—C12	1.388 (6)	C42—H42C	0.9800
C11—C14	1.498 (6)	C43—O1	1.252 (5)
C12—C13	1.381 (6)	C43—O2	1.263 (6)
C12—H12	0.9500	C43—C44	1.507 (6)
C13—H13	0.9500	C44—C45	1.445 (8)
C14—H14A	0.9800	C44—C45A	1.466 (11)
C14—H14B	0.9800	C44—H44A	0.9600
C14—H14C	0.9800	C44—H44B	0.9600
C15—C16	1.393 (6)	C44—H44C	0.9601
C15—C20	1.398 (6)	C44—H44D	0.9599
C15—P1	1.820 (4)	C45—H45A	0.9800
C16—C17	1.388 (6)	C45—H45B	0.9800
C16—H16	0.9500	C45—H45C	0.9800
C17—C18	1.385 (7)	C45A—H45D	0.9800
C17—H17	0.9500	C45A—H45E	0.9800
C18—C19	1.400 (7)	C45A—H45F	0.9800
C18—C21	1.514 (6)	C46—O4	1.262 (5)
C19—C20	1.385 (6)	C46—O3	1.267 (5)
C19—H19	0.9500	C46—C47	1.505 (6)
C20—H20	0.9500	C47—C48	1.513 (7)
C21—H21A	0.9800	C47—H47A	0.9900
C21—H21B	0.9800	C47—H47B	0.9900
C21—H21C	0.9800	C48—H48A	0.9800
C22—C23	1.388 (6)	C48—H48B	0.9800

C22—C27	1.392 (6)	C48—H48C	0.9800
C22—P2	1.831 (4)	C49—O5	1.143 (5)
C23—C24	1.394 (6)	C49—Os1	1.866 (5)
C23—H23	0.9500	C50—O6	1.162 (5)
C24—C25	1.394 (7)	C50—Os1	1.849 (5)
C24—H24	0.9500	C51—O7	1.161 (5)
C25—C26	1.395 (7)	C51—Os2	1.853 (5)
C25—C28	1.509 (6)	C52—O8	1.168 (5)
C26—C27	1.392 (6)	C52—Os2	1.843 (5)
C26—H26	0.9500	C53—Cl1	1.751 (6)
C27—H27	0.9500	C53—Cl2	1.753 (6)
C28—H28A	0.9800	C53—H53A	0.9900
C28—H28B	0.9800	C53—H53B	0.9900
C28—H28C	0.9800	O1—Os1	2.127 (3)
C29—C34	1.389 (6)	O2—Os2	2.110 (3)
C29—C30	1.395 (6)	O3—Os1	2.118 (3)
C29—P2	1.836 (4)	O4—Os2	2.132 (3)
C30—C31	1.377 (6)	Os1—P1	2.4037 (11)
C30—H30	0.9500	Os1—Os2	2.7677 (3)
C31—C32	1.403 (7)	Os2—P2	2.4022 (12)
C31—H31	0.9500		

C6—C1—C2	118.1 (4)	C41—C36—P2	121.7 (3)
C6—C1—P1	120.6 (3)	C37—C36—P2	119.5 (3)
C2—C1—P1	121.3 (3)	C38—C37—C36	119.9 (4)
C1—C2—C3	120.8 (4)	C38—C37—H37	120.1
C1—C2—H2	119.6	C36—C37—H37	120.1
C3—C2—H2	119.6	C37—C38—C39	121.5 (5)
C4—C3—C2	121.3 (4)	C37—C38—H38	119.3
C4—C3—H3	119.4	C39—C38—H38	119.3
C2—C3—H3	119.4	C40—C39—C38	117.8 (4)
C5—C4—C3	117.4 (4)	C40—C39—C42	121.5 (5)
C5—C4—C7	121.4 (4)	C38—C39—C42	120.6 (5)
C3—C4—C7	121.2 (4)	C39—C40—C41	121.5 (4)
C4—C5—C6	121.6 (4)	C39—C40—H40	119.2
C4—C5—H5	119.2	C41—C40—H40	119.2
C6—C5—H5	119.2	C40—C41—C36	120.7 (4)
C1—C6—C5	120.7 (4)	C40—C41—H41	119.6
C1—C6—H6	119.6	C36—C41—H41	119.6
C5—C6—H6	119.6	C39—C42—H42A	109.5
C4—C7—H7A	109.5	C39—C42—H42B	109.5
C4—C7—H7B	109.5	H42A—C42—H42B	109.5
H7A—C7—H7B	109.5	C39—C42—H42C	109.5
C4—C7—H7C	109.5	H42A—C42—H42C	109.5
H7A—C7—H7C	109.5	H42B—C42—H42C	109.5
H7B—C7—H7C	109.5	O1—C43—O2	125.7 (4)
C9—C8—C13	117.5 (4)	O1—C43—C44	117.9 (4)
C9—C8—P1	122.2 (3)	O2—C43—C44	116.4 (4)

C13—C8—P1	120.1 (3)	C45—C44—C43	115.1 (5)
C8—C9—C10	120.4 (4)	C45A—C44—C43	112.9 (7)
C8—C9—H9	119.8	C45—C44—H44A	108.4
C10—C9—H9	119.8	C43—C44—H44A	108.6
C11—C10—C9	121.8 (4)	C45—C44—H44B	108.6
C11—C10—H10	119.1	C43—C44—H44B	108.4
C9—C10—H10	119.1	H44A—C44—H44B	107.5
C10—C11—C12	117.2 (4)	C45A—C44—H44C	109.4
C10—C11—C14	120.4 (4)	C43—C44—H44C	108.9
C12—C11—C14	122.3 (4)	C45A—C44—H44D	108.7
C13—C12—C11	121.7 (4)	C43—C44—H44D	109.1
C13—C12—H12	119.2	H44C—C44—H44D	107.8
C11—C12—H12	119.2	C44—C45—H45A	109.5
C12—C13—C8	121.3 (4)	C44—C45—H45B	109.5
C12—C13—H13	119.4	H45A—C45—H45B	109.5
C8—C13—H13	119.4	C44—C45—H45C	109.5
C11—C14—H14A	109.5	H45A—C45—H45C	109.5
C11—C14—H14B	109.5	H45B—C45—H45C	109.5
H14A—C14—H14B	109.5	C44—C45A—H45D	109.5
C11—C14—H14C	109.5	C44—C45A—H45E	109.5
H14A—C14—H14C	109.5	H45D—C45A—H45E	109.5
H14B—C14—H14C	109.5	C44—C45A—H45F	109.5
C16—C15—C20	117.7 (4)	H45D—C45A—H45F	109.5
C16—C15—P1	122.8 (3)	H45E—C45A—H45F	109.5
C20—C15—P1	119.1 (3)	O4—C46—O3	125.3 (4)
C17—C16—C15	121.1 (4)	O4—C46—C47	118.6 (4)
C17—C16—H16	119.5	O3—C46—C47	116.1 (4)
C15—C16—H16	119.5	C46—C47—C48	110.3 (4)
C18—C17—C16	121.2 (4)	C46—C47—H47A	109.6
C18—C17—H17	119.4	C48—C47—H47A	109.6
C16—C17—H17	119.4	C46—C47—H47B	109.6
C17—C18—C19	118.0 (4)	C48—C47—H47B	109.6
C17—C18—C21	121.2 (4)	H47A—C47—H47B	108.1
C19—C18—C21	120.8 (4)	C47—C48—H48A	109.5
C20—C19—C18	120.9 (4)	C47—C48—H48B	109.5
C20—C19—H19	119.6	H48A—C48—H48B	109.5
C18—C19—H19	119.6	C47—C48—H48C	109.5
C19—C20—C15	121.0 (4)	H48A—C48—H48C	109.5
C19—C20—H20	119.5	H48B—C48—H48C	109.5
C15—C20—H20	119.5	O5—C49—Os1	179.3 (4)
C18—C21—H21A	109.5	O6—C50—Os1	179.2 (4)
C18—C21—H21B	109.5	O7—C51—Os2	178.1 (4)
H21A—C21—H21B	109.5	O8—C52—Os2	179.0 (4)
C18—C21—H21C	109.5	Cl1—C53—Cl2	112.5 (3)
H21A—C21—H21C	109.5	Cl1—C53—H53A	109.1
H21B—C21—H21C	109.5	Cl2—C53—H53A	109.1
C23—C22—C27	118.5 (4)	Cl1—C53—H53B	109.1
C23—C22—P2	123.5 (3)	Cl2—C53—H53B	109.1

C27—C22—P2	117.7 (3)	H53A—C53—H53B	107.8
C22—C23—C24	120.9 (4)	C43—O1—Os1	122.8 (3)
C22—C23—H23	119.5	C43—O2—Os2	125.0 (3)
C24—C23—H23	119.5	C46—O3—Os1	126.2 (3)
C23—C24—C25	120.7 (4)	C46—O4—Os2	121.5 (3)
C23—C24—H24	119.7	C50—Os1—C49	89.3 (2)
C25—C24—H24	119.7	C50—Os1—O3	176.98 (17)
C24—C25—C26	118.2 (4)	C49—Os1—O3	93.43 (17)
C24—C25—C28	120.9 (4)	C50—Os1—O1	95.18 (17)
C26—C25—C28	120.8 (4)	C49—Os1—O1	175.29 (17)
C27—C26—C25	120.8 (4)	O3—Os1—O1	82.10 (13)
C27—C26—H26	119.6	C50—Os1—P1	94.55 (13)
C25—C26—H26	119.6	C49—Os1—P1	96.01 (14)
C26—C27—C22	120.7 (4)	O3—Os1—P1	86.56 (9)
C26—C27—H27	119.6	O1—Os1—P1	85.24 (9)
C22—C27—H27	119.6	C50—Os1—Os2	97.03 (13)
C25—C28—H28A	109.5	C49—Os1—Os2	94.85 (14)
C25—C28—H28B	109.5	O3—Os1—Os2	81.38 (8)
H28A—C28—H28B	109.5	O1—Os1—Os2	83.06 (8)
C25—C28—H28C	109.5	P1—Os1—Os2	164.21 (3)
H28A—C28—H28C	109.5	C52—Os2—C51	90.2 (2)
H28B—C28—H28C	109.5	C52—Os2—O2	95.23 (16)
C34—C29—C30	118.2 (4)	C51—Os2—O2	174.03 (17)
C34—C29—P2	122.6 (3)	C52—Os2—O4	174.58 (15)
C30—C29—P2	119.1 (3)	C51—Os2—O4	93.70 (17)
C31—C30—C29	121.2 (4)	O2—Os2—O4	80.69 (13)
C31—C30—H30	119.4	C52—Os2—P2	93.35 (13)
C29—C30—H30	119.4	C51—Os2—P2	97.51 (14)
C30—C31—C32	120.8 (4)	O2—Os2—P2	84.59 (9)
C30—C31—H31	119.6	O4—Os2—P2	89.83 (9)
C32—C31—H31	119.6	C52—Os2—Os1	91.74 (13)
C33—C32—C31	117.9 (4)	C51—Os2—Os1	95.39 (13)
C33—C32—C35	120.9 (5)	O2—Os2—Os1	82.07 (9)
C31—C32—C35	121.2 (4)	O4—Os2—Os1	84.19 (8)
C32—C33—C34	121.4 (5)	P2—Os2—Os1	166.10 (3)
C32—C33—H33	119.3	C8—P1—C15	103.4 (2)
C34—C33—H33	119.3	C8—P1—C1	101.52 (19)
C29—C34—C33	120.5 (4)	C15—P1—C1	103.44 (19)
C29—C34—H34	119.8	C8—P1—Os1	116.39 (14)
C33—C34—H34	119.8	C15—P1—Os1	112.59 (14)
C32—C35—H35A	109.5	C1—P1—Os1	117.63 (14)
C32—C35—H35B	109.5	C36—P2—C22	104.9 (2)
H35A—C35—H35B	109.5	C36—P2—C29	102.2 (2)
C32—C35—H35C	109.5	C22—P2—C29	101.4 (2)
H35A—C35—H35C	109.5	C36—P2—Os2	111.30 (15)
H35B—C35—H35C	109.5	C22—P2—Os2	117.41 (15)
C41—C36—C37	118.4 (4)	C29—P2—Os2	117.74 (15)

C6—C1—C2—C3	1.6 (7)	C38—C39—C40—C41	−0.5 (7)
P1—C1—C2—C3	−177.7 (4)	C42—C39—C40—C41	−179.0 (5)
C1—C2—C3—C4	0.2 (7)	C39—C40—C41—C36	−1.6 (7)
C2—C3—C4—C5	−1.9 (7)	C37—C36—C41—C40	1.7 (7)
C2—C3—C4—C7	177.0 (4)	P2—C36—C41—C40	174.1 (4)
C3—C4—C5—C6	1.9 (7)	O1—C43—C44—C45	−4.3 (8)
C7—C4—C5—C6	−177.0 (4)	O2—C43—C44—C45	177.7 (6)
C2—C1—C6—C5	−1.6 (7)	O1—C43—C44—C45A	50.5 (14)
P1—C1—C6—C5	177.7 (4)	O2—C43—C44—C45A	−127.6 (14)
C4—C5—C6—C1	−0.2 (7)	O4—C46—C47—C48	107.9 (5)
C13—C8—C9—C10	−2.1 (6)	O3—C46—C47—C48	−71.4 (5)
P1—C8—C9—C10	173.0 (3)	O2—C43—O1—Os1	10.8 (7)
C8—C9—C10—C11	2.5 (7)	C44—C43—O1—Os1	−167.0 (3)
C9—C10—C11—C12	−0.6 (7)	O1—C43—O2—Os2	0.5 (7)
C9—C10—C11—C14	−177.8 (4)	C44—C43—O2—Os2	178.4 (3)
C10—C11—C12—C13	−1.6 (7)	O4—C46—O3—Os1	−5.9 (6)
C14—C11—C12—C13	175.6 (4)	C47—C46—O3—Os1	173.4 (3)
C11—C12—C13—C8	2.0 (7)	O3—C46—O4—Os2	14.4 (6)
C9—C8—C13—C12	−0.1 (6)	C47—C46—O4—Os2	−164.8 (3)
P1—C8—C13—C12	−175.3 (3)	C9—C8—P1—C15	27.8 (4)
C20—C15—C16—C17	−2.3 (7)	C13—C8—P1—C15	−157.2 (4)
P1—C15—C16—C17	−175.6 (4)	C9—C8—P1—C1	−79.2 (4)
C15—C16—C17—C18	0.5 (7)	C13—C8—P1—C1	95.8 (4)
C16—C17—C18—C19	0.5 (7)	C9—C8—P1—Os1	151.8 (3)
C16—C17—C18—C21	−179.1 (5)	C13—C8—P1—Os1	−33.3 (4)
C17—C18—C19—C20	0.5 (7)	C16—C15—P1—C8	−132.7 (4)
C21—C18—C19—C20	180.0 (4)	C20—C15—P1—C8	54.1 (4)
C18—C19—C20—C15	−2.4 (7)	C16—C15—P1—C1	−27.2 (4)
C16—C15—C20—C19	3.2 (7)	C20—C15—P1—C1	159.7 (3)
P1—C15—C20—C19	176.8 (3)	C16—C15—P1—Os1	100.9 (4)
C27—C22—C23—C24	1.3 (7)	C20—C15—P1—Os1	−72.3 (4)
P2—C22—C23—C24	−172.8 (4)	C6—C1—P1—C8	46.7 (4)
C22—C23—C24—C25	0.8 (7)	C2—C1—P1—C8	−134.1 (4)
C23—C24—C25—C26	−2.7 (7)	C6—C1—P1—C15	−60.3 (4)
C23—C24—C25—C28	176.8 (4)	C2—C1—P1—C15	118.9 (4)
C24—C25—C26—C27	2.5 (7)	C6—C1—P1—Os1	174.9 (3)
C28—C25—C26—C27	−176.9 (4)	C2—C1—P1—Os1	−5.9 (4)
C25—C26—C27—C22	−0.5 (7)	C41—C36—P2—C22	130.3 (4)
C23—C22—C27—C26	−1.5 (7)	C37—C36—P2—C22	−57.4 (4)
P2—C22—C27—C26	173.0 (3)	C41—C36—P2—C29	24.9 (4)
C34—C29—C30—C31	1.5 (7)	C37—C36—P2—C29	−162.8 (4)
P2—C29—C30—C31	−177.7 (4)	C41—C36—P2—Os2	−101.7 (4)
C29—C30—C31—C32	−2.3 (7)	C37—C36—P2—Os2	70.6 (4)
C30—C31—C32—C33	1.6 (7)	C23—C22—P2—C36	−16.3 (4)
C30—C31—C32—C35	−178.9 (5)	C27—C22—P2—C36	169.6 (3)
C31—C32—C33—C34	−0.4 (7)	C23—C22—P2—C29	89.8 (4)
C35—C32—C33—C34	−179.9 (5)	C27—C22—P2—C29	−84.3 (4)
C30—C29—C34—C33	−0.3 (7)	C23—C22—P2—Os2	−140.4 (3)

P2—C29—C34—C33	178.9 (4)	C27—C22—P2—Os2	45.4 (4)
C32—C33—C34—C29	−0.3 (7)	C34—C29—P2—C36	−120.5 (4)
C41—C36—C37—C38	0.3 (7)	C30—C29—P2—C36	58.7 (4)
P2—C36—C37—C38	−172.3 (4)	C34—C29—P2—C22	131.3 (4)
C36—C37—C38—C39	−2.4 (8)	C30—C29—P2—C22	−49.5 (4)
C37—C38—C39—C40	2.5 (8)	C34—C29—P2—Os2	1.8 (4)
C37—C38—C39—C42	−179.0 (5)	C30—C29—P2—Os2	−179.0 (3)

Bis(*μ*-formato-1*κO*:2*κO′*)bis[tris(4-methylphenyl)phosphane]-1*κP*,2*κP*-bis(dicarbonylosmium)(*Os—Os*) (8)

Crystal data

$[Os_2(CHO_2)_2(C_{21}H_{21}P)_2(CO)_4]$
M_r = 1191.17
Triclinic, $P\overline{1}$
a = 10.7707 (4) Å
b = 10.8014 (4) Å
c = 20.3790 (7) Å
α = 104.393 (1)°
β = 91.009 (1)°
γ = 98.993 (1)°
V = 2264.27 (14) Å^3
Z = 2
$F(000)$ = 1156
D_x = 1.747 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9955 reflections
θ = 2.5–27.1°
μ = 5.73 mm^{-1}
T = 200 K
Block, colorless
0.15 × 0.11 × 0.04 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2008)
T_{min} = 0.538, T_{max} = 0.746
30910 measured reflections
9960 independent reflections
8554 reflections with $I > 2\sigma(I)$
R_{int} = 0.058
θ_{max} = 27.1°, θ_{min} = 1.0°
h = −13→13
k = −13→13
l = −26→26

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.082
S = 1.06
9960 reflections
548 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0249P)^2 + 1.5967P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 1.19 e Å^{-3}
$\Delta\rho_{min}$ = −1.09 e Å^{-3}
Extinction correction: SHELXL2018 (Sheldrick, 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00032 (9)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
Os1	0.69417 (2)	0.59590 (2)	0.29383 (2)	0.02493 (6)

P1	0.81773 (10)	0.76444 (10)	0.38172 (5)	0.0238 (2)
O1	0.6478 (4)	0.7406 (3)	0.24699 (18)	0.0461 (9)
C1	0.5899 (6)	0.7107 (5)	0.1902 (3)	0.0508 (14)
H1	0.587681	0.779927	0.169394	0.061*
Os2	0.50480 (2)	0.44535 (2)	0.20412 (2)	0.02496 (6)
O2	0.5349 (3)	0.6028 (3)	0.15726 (16)	0.0386 (8)
P2	0.32402 (10)	0.36297 (11)	0.12322 (5)	0.0254 (2)
C2	0.4312 (5)	0.6229 (5)	0.3295 (2)	0.0422 (12)
H2	0.371286	0.660669	0.358300	0.051*
C3	0.8164 (5)	0.5635 (5)	0.2324 (3)	0.0435 (12)
O3	0.5407 (3)	0.6400 (3)	0.35564 (16)	0.0374 (8)
C4	0.7314 (5)	0.4638 (5)	0.3293 (3)	0.0392 (11)
O4	0.3907 (3)	0.5630 (3)	0.27093 (16)	0.0389 (8)
O5	0.8900 (4)	0.5444 (5)	0.1927 (2)	0.0729 (15)
C5	0.6153 (5)	0.3528 (5)	0.1529 (2)	0.0381 (11)
O6	0.7552 (4)	0.3798 (4)	0.3495 (2)	0.0662 (13)
C6	0.4812 (5)	0.3237 (5)	0.2534 (3)	0.0408 (12)
O7	0.6866 (4)	0.2952 (4)	0.1227 (2)	0.0571 (11)
C7	0.7472 (4)	0.9097 (4)	0.4120 (2)	0.0261 (9)
C8	0.7160 (4)	0.9755 (4)	0.3650 (2)	0.0302 (9)
H8	0.730434	0.943097	0.318522	0.036*
O8	0.4676 (5)	0.2497 (4)	0.2865 (2)	0.0745 (15)
C9	0.6641 (4)	1.0875 (4)	0.3851 (2)	0.0317 (10)
H9	0.645251	1.131966	0.352375	0.038*
C10	0.6394 (5)	1.1354 (5)	0.4520 (3)	0.0383 (11)
C11	0.6713 (6)	1.0704 (5)	0.4985 (3)	0.0531 (15)
H11	0.656524	1.103077	0.544958	0.064*
C12	0.7240 (6)	0.9594 (5)	0.4795 (2)	0.0449 (13)
H12	0.744483	0.916591	0.512723	0.054*
C13	0.9740 (4)	0.8264 (4)	0.3597 (2)	0.0266 (9)
C14	1.0236 (4)	0.9585 (4)	0.3734 (2)	0.0325 (10)
H14	0.975167	1.021349	0.395397	0.039*
C15	1.1443 (5)	0.9977 (5)	0.3546 (3)	0.0400 (11)
H15	1.176412	1.087678	0.363518	0.048*
C16	1.2181 (5)	0.9099 (6)	0.3235 (3)	0.0424 (12)
C17	1.1687 (5)	0.7792 (5)	0.3113 (2)	0.0393 (11)
H17	1.219089	0.716909	0.290980	0.047*
C18	1.0491 (4)	0.7374 (5)	0.3278 (2)	0.0339 (10)
H18	1.017219	0.647215	0.317486	0.041*
C19	0.8480 (4)	0.7134 (4)	0.4589 (2)	0.0266 (9)
C20	0.7464 (5)	0.6515 (5)	0.4866 (2)	0.0361 (10)
H20	0.665145	0.634224	0.464179	0.043*
C21	0.7618 (5)	0.6148 (5)	0.5460 (3)	0.0416 (12)
H21	0.690740	0.575966	0.564927	0.050*
C22	0.8811 (5)	0.6343 (5)	0.5784 (3)	0.0430 (12)
C23	0.9822 (5)	0.6931 (5)	0.5503 (3)	0.0438 (12)
H23	1.064031	0.706989	0.571739	0.053*
C24	0.9663 (4)	0.7326 (4)	0.4905 (2)	0.0334 (10)

H24	1.037121	0.772633	0.471843	0.040*
C25	0.2555 (4)	0.4836 (4)	0.0925 (2)	0.0294 (9)
C26	0.2262 (5)	0.4730 (5)	0.0242 (2)	0.0369 (11)
H26	0.244275	0.400683	−0.009421	0.044*
C27	0.1711 (5)	0.5664 (5)	0.0050 (3)	0.0439 (12)
H27	0.152324	0.557505	−0.041832	0.053*
C28	0.1428 (5)	0.6716 (5)	0.0515 (3)	0.0409 (12)
C29	0.1717 (5)	0.6832 (5)	0.1202 (3)	0.0464 (13)
H29	0.152913	0.755572	0.153414	0.056*
C30	0.2271 (5)	0.5909 (5)	0.1400 (3)	0.0432 (12)
H30	0.246246	0.600576	0.186823	0.052*
C31	0.1902 (4)	0.2697 (4)	0.1517 (2)	0.0290 (9)
C32	0.2086 (4)	0.1642 (5)	0.1759 (2)	0.0347 (10)
H32	0.291382	0.145594	0.179533	0.042*
C33	0.1090 (5)	0.0854 (6)	0.1951 (3)	0.0460 (13)
H33	0.123817	0.012699	0.210535	0.055*
C34	−0.0121 (5)	0.1127 (8)	0.1916 (3)	0.0634 (19)
C35	−0.0309 (5)	0.2184 (8)	0.1704 (3)	0.067 (2)
H35	−0.113138	0.239727	0.169983	0.080*
C36	0.0684 (5)	0.2966 (6)	0.1491 (3)	0.0458 (13)
H36	0.052460	0.368229	0.132960	0.055*
C37	0.3638 (4)	0.2534 (4)	0.0457 (2)	0.0277 (9)
C38	0.2969 (5)	0.1299 (5)	0.0181 (2)	0.0383 (11)
H38	0.222740	0.100150	0.037977	0.046*
C39	0.3387 (6)	0.0496 (5)	−0.0390 (3)	0.0464 (13)
H39	0.292376	−0.034764	−0.057637	0.056*
C40	0.4462 (5)	0.0907 (5)	−0.0687 (2)	0.0386 (11)
C41	0.5085 (5)	0.2152 (5)	−0.0420 (2)	0.0388 (11)
H41	0.580414	0.246334	−0.063274	0.047*
C42	0.4697 (4)	0.2957 (5)	0.0144 (2)	0.0355 (10)
H42	0.515520	0.380576	0.032129	0.043*
C43	0.5805 (7)	1.2550 (6)	0.4731 (3)	0.0644 (18)
H43A	0.597271	1.292723	0.521960	0.097*
H43B	0.489365	1.232428	0.462586	0.097*
H43C	0.616319	1.318136	0.448422	0.097*
C44	1.3501 (6)	0.9517 (7)	0.3040 (4)	0.0677 (19)
H44A	1.408076	0.903532	0.320817	0.102*
H44B	1.376363	1.044780	0.324159	0.102*
H44C	1.351387	0.933956	0.254512	0.102*
C45	0.8984 (7)	0.5927 (7)	0.6431 (3)	0.0692 (19)
H45A	0.988432	0.598946	0.654205	0.104*
H45B	0.856481	0.502846	0.636818	0.104*
H45C	0.861447	0.649194	0.680233	0.104*
C46	0.0825 (7)	0.7754 (6)	0.0301 (3)	0.0637 (18)
H46A	0.115889	0.787900	−0.012636	0.096*
H46B	0.101759	0.857079	0.065351	0.096*
H46C	−0.008993	0.747778	0.023794	0.096*
C47	−0.1216 (6)	0.0281 (11)	0.2136 (5)	0.118 (4)

H47A	−0.199251	0.030386	0.188434	0.177*
H47B	−0.130619	0.060599	0.262317	0.177*
H47C	−0.105507	−0.061321	0.204095	0.177*
C48	0.4942 (7)	0.0012 (6)	−0.1291 (3)	0.0611 (17)
H48A	0.511144	−0.076731	−0.116634	0.092*
H48B	0.571967	0.046052	−0.142700	0.092*
H48C	0.430502	−0.023364	−0.167034	0.092*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Os1	0.02462 (10)	0.02413 (10)	0.02483 (10)	0.00310 (7)	−0.00128 (6)	0.00475 (7)
P1	0.0245 (5)	0.0219 (5)	0.0241 (5)	0.0042 (4)	−0.0020 (4)	0.0041 (4)
O1	0.065 (3)	0.0304 (18)	0.040 (2)	0.0011 (17)	−0.0208 (18)	0.0111 (15)
C1	0.066 (4)	0.040 (3)	0.048 (3)	−0.005 (3)	−0.016 (3)	0.024 (3)
Os2	0.02504 (10)	0.02498 (10)	0.02367 (10)	0.00369 (7)	−0.00202 (6)	0.00448 (7)
O2	0.046 (2)	0.0348 (18)	0.0338 (18)	−0.0027 (15)	−0.0078 (15)	0.0124 (14)
P2	0.0238 (5)	0.0291 (6)	0.0215 (5)	0.0043 (4)	−0.0015 (4)	0.0034 (4)
C2	0.035 (3)	0.055 (3)	0.033 (3)	0.012 (2)	0.003 (2)	0.002 (2)
C3	0.034 (3)	0.045 (3)	0.041 (3)	−0.002 (2)	0.003 (2)	−0.003 (2)
O3	0.0302 (18)	0.050 (2)	0.0273 (16)	0.0086 (15)	−0.0027 (13)	−0.0003 (15)
C4	0.038 (3)	0.033 (3)	0.046 (3)	0.006 (2)	−0.008 (2)	0.008 (2)
O4	0.0290 (17)	0.052 (2)	0.0298 (17)	0.0134 (15)	−0.0041 (13)	−0.0032 (15)
O5	0.040 (2)	0.105 (4)	0.050 (2)	−0.002 (2)	0.0157 (19)	−0.017 (2)
C5	0.035 (3)	0.037 (3)	0.037 (3)	0.009 (2)	−0.010 (2)	−0.002 (2)
O6	0.075 (3)	0.046 (2)	0.086 (3)	0.014 (2)	−0.026 (2)	0.031 (2)
C6	0.041 (3)	0.039 (3)	0.039 (3)	−0.007 (2)	−0.011 (2)	0.011 (2)
O7	0.038 (2)	0.060 (3)	0.063 (3)	0.0250 (19)	−0.0022 (18)	−0.011 (2)
C7	0.023 (2)	0.026 (2)	0.027 (2)	0.0043 (16)	−0.0026 (16)	0.0022 (17)
C8	0.037 (3)	0.030 (2)	0.026 (2)	0.0077 (19)	0.0042 (18)	0.0085 (18)
O8	0.098 (4)	0.056 (3)	0.068 (3)	−0.028 (2)	−0.035 (3)	0.041 (2)
C9	0.038 (3)	0.029 (2)	0.032 (2)	0.0123 (19)	0.0020 (19)	0.0114 (18)
C10	0.044 (3)	0.034 (3)	0.039 (3)	0.015 (2)	0.012 (2)	0.007 (2)
C11	0.089 (5)	0.050 (3)	0.026 (2)	0.034 (3)	0.016 (3)	0.007 (2)
C12	0.072 (4)	0.042 (3)	0.027 (2)	0.027 (3)	0.008 (2)	0.009 (2)
C13	0.030 (2)	0.026 (2)	0.021 (2)	0.0042 (17)	−0.0055 (16)	0.0015 (16)
C14	0.029 (2)	0.030 (2)	0.037 (2)	0.0031 (18)	−0.0066 (19)	0.0070 (19)
C15	0.031 (3)	0.041 (3)	0.049 (3)	−0.005 (2)	−0.008 (2)	0.021 (2)
C16	0.030 (3)	0.063 (4)	0.041 (3)	0.005 (2)	0.001 (2)	0.028 (3)
C17	0.030 (2)	0.053 (3)	0.037 (3)	0.016 (2)	0.003 (2)	0.009 (2)
C18	0.030 (2)	0.033 (2)	0.037 (3)	0.0085 (19)	−0.0021 (19)	0.004 (2)
C19	0.030 (2)	0.023 (2)	0.025 (2)	0.0031 (17)	−0.0061 (17)	0.0041 (16)
C20	0.035 (3)	0.038 (3)	0.037 (3)	0.002 (2)	−0.003 (2)	0.016 (2)
C21	0.049 (3)	0.036 (3)	0.040 (3)	0.000 (2)	0.001 (2)	0.013 (2)
C22	0.058 (3)	0.038 (3)	0.037 (3)	0.012 (2)	−0.004 (2)	0.014 (2)
C23	0.045 (3)	0.045 (3)	0.042 (3)	0.010 (2)	−0.016 (2)	0.012 (2)
C24	0.034 (2)	0.034 (2)	0.034 (2)	0.0031 (19)	−0.0024 (19)	0.0115 (19)
C25	0.030 (2)	0.031 (2)	0.026 (2)	0.0054 (18)	−0.0033 (17)	0.0055 (18)

C26	0.042 (3)	0.034 (2)	0.031 (2)	0.004 (2)	−0.007 (2)	0.0045 (19)
C27	0.050 (3)	0.046 (3)	0.036 (3)	0.000 (2)	−0.006 (2)	0.016 (2)
C28	0.037 (3)	0.040 (3)	0.046 (3)	−0.003 (2)	−0.011 (2)	0.018 (2)
C29	0.054 (3)	0.045 (3)	0.041 (3)	0.019 (3)	−0.007 (2)	0.006 (2)
C30	0.050 (3)	0.048 (3)	0.031 (3)	0.021 (2)	−0.006 (2)	0.003 (2)
C31	0.029 (2)	0.037 (2)	0.021 (2)	0.0067 (19)	0.0039 (17)	0.0058 (18)
C32	0.030 (2)	0.042 (3)	0.030 (2)	0.004 (2)	0.0009 (18)	0.007 (2)
C33	0.041 (3)	0.063 (4)	0.036 (3)	−0.005 (2)	0.000 (2)	0.023 (2)
C34	0.035 (3)	0.111 (6)	0.053 (4)	−0.005 (3)	0.001 (3)	0.049 (4)
C35	0.025 (3)	0.130 (6)	0.065 (4)	0.015 (3)	0.007 (3)	0.060 (4)
C36	0.031 (3)	0.076 (4)	0.041 (3)	0.014 (3)	0.003 (2)	0.030 (3)
C37	0.027 (2)	0.031 (2)	0.023 (2)	0.0040 (18)	−0.0016 (16)	0.0051 (17)
C38	0.042 (3)	0.037 (3)	0.029 (2)	−0.002 (2)	0.009 (2)	0.001 (2)
C39	0.068 (4)	0.031 (3)	0.035 (3)	0.006 (2)	0.006 (2)	0.000 (2)
C40	0.053 (3)	0.043 (3)	0.024 (2)	0.018 (2)	0.010 (2)	0.009 (2)
C41	0.031 (3)	0.057 (3)	0.032 (2)	0.011 (2)	0.0092 (19)	0.014 (2)
C42	0.034 (3)	0.040 (3)	0.029 (2)	0.001 (2)	0.0024 (19)	0.005 (2)
C43	0.090 (5)	0.056 (4)	0.057 (4)	0.044 (4)	0.021 (3)	0.012 (3)
C44	0.039 (3)	0.093 (5)	0.080 (5)	0.001 (3)	0.014 (3)	0.045 (4)
C45	0.084 (5)	0.080 (5)	0.059 (4)	0.015 (4)	−0.010 (3)	0.045 (4)
C46	0.074 (5)	0.048 (3)	0.074 (4)	0.014 (3)	−0.017 (4)	0.025 (3)
C47	0.036 (4)	0.210 (11)	0.146 (8)	−0.016 (5)	0.000 (4)	0.138 (8)
C48	0.085 (5)	0.061 (4)	0.044 (3)	0.039 (4)	0.020 (3)	0.007 (3)

Geometric parameters (Å, º)

Os1—P1	2.4148 (11)	C22—C45	1.512 (7)
Os1—O1	2.138 (3)	C23—H23	0.9500
Os1—Os2	2.7387 (2)	C23—C24	1.403 (6)
Os1—C3	1.845 (5)	C24—H24	0.9500
Os1—O3	2.137 (3)	C25—C26	1.395 (6)
Os1—C4	1.844 (5)	C25—C30	1.392 (6)
P1—C7	1.825 (4)	C26—H26	0.9500
P1—C13	1.818 (5)	C26—C27	1.378 (7)
P1—C19	1.831 (4)	C27—H27	0.9500
O1—C1	1.248 (6)	C27—C28	1.365 (8)
C1—H1	0.9500	C28—C29	1.397 (7)
C1—O2	1.242 (6)	C28—C46	1.528 (7)
Os2—O2	2.134 (3)	C29—H29	0.9500
Os2—P2	2.4325 (11)	C29—C30	1.374 (7)
Os2—O4	2.160 (3)	C30—H30	0.9500
Os2—C5	1.844 (5)	C31—C32	1.390 (6)
Os2—C6	1.834 (5)	C31—C36	1.390 (6)
P2—C25	1.824 (4)	C32—H32	0.9500
P2—C31	1.815 (5)	C32—C33	1.385 (7)
P2—C37	1.826 (4)	C33—H33	0.9500
C2—H2	0.9500	C33—C34	1.386 (8)
C2—O3	1.253 (6)	C34—C35	1.359 (9)

C2—O4	1.243 (6)	C34—C47	1.520 (8)
C3—O5	1.148 (6)	C35—H35	0.9500
C4—O6	1.145 (6)	C35—C36	1.400 (8)
C5—O7	1.155 (6)	C36—H36	0.9500
C6—O8	1.161 (6)	C37—C38	1.388 (6)
C7—C8	1.390 (6)	C37—C42	1.387 (6)
C7—C12	1.390 (6)	C38—H38	0.9500
C8—H8	0.9500	C38—C39	1.395 (7)
C8—C9	1.384 (6)	C39—H39	0.9500
C9—H9	0.9500	C39—C40	1.379 (7)
C9—C10	1.378 (6)	C40—C41	1.377 (7)
C10—C11	1.381 (7)	C40—C48	1.518 (7)
C10—C43	1.499 (7)	C41—H41	0.9500
C11—H11	0.9500	C41—C42	1.374 (6)
C11—C12	1.378 (7)	C42—H42	0.9500
C12—H12	0.9500	C43—H43A	0.9800
C13—C14	1.400 (6)	C43—H43B	0.9800
C13—C18	1.397 (6)	C43—H43C	0.9800
C14—H14	0.9500	C44—H44A	0.9800
C14—C15	1.393 (7)	C44—H44B	0.9800
C15—H15	0.9500	C44—H44C	0.9800
C15—C16	1.374 (8)	C45—H45A	0.9800
C16—C17	1.387 (8)	C45—H45B	0.9800
C16—C44	1.514 (7)	C45—H45C	0.9800
C17—H17	0.9500	C46—H46A	0.9800
C17—C18	1.373 (7)	C46—H46B	0.9800
C18—H18	0.9500	C46—H46C	0.9800
C19—C20	1.394 (6)	C47—H47A	0.9800
C19—C24	1.381 (6)	C47—H47B	0.9800
C20—H20	0.9500	C47—H47C	0.9800
C20—C21	1.378 (7)	C48—H48A	0.9800
C21—H21	0.9500	C48—H48B	0.9800
C21—C22	1.396 (7)	C48—H48C	0.9800
C22—C23	1.380 (8)		

P1—Os1—Os2	164.48 (3)	C22—C21—H21	119.7
O1—Os1—P1	89.31 (9)	C21—C22—C45	120.6 (5)
O1—Os1—Os2	81.48 (9)	C23—C22—C21	118.4 (5)
C3—Os1—P1	99.28 (16)	C23—C22—C45	121.0 (5)
C3—Os1—O1	89.2 (2)	C22—C23—H23	119.4
C3—Os1—Os2	93.09 (16)	C22—C23—C24	121.2 (5)
C3—Os1—O3	173.12 (19)	C24—C23—H23	119.4
O3—Os1—P1	84.04 (9)	C19—C24—C23	120.1 (5)
O3—Os1—O1	84.79 (15)	C19—C24—H24	120.0
O3—Os1—Os2	82.71 (8)	C23—C24—H24	120.0
C4—Os1—P1	93.97 (15)	C26—C25—P2	123.9 (4)
C4—Os1—O1	176.54 (17)	C30—C25—P2	118.3 (3)
C4—Os1—Os2	95.56 (15)	C30—C25—C26	117.8 (4)

C4—Os1—C3	89.2 (2)	C25—C26—H26	119.8
C4—Os1—O3	96.7 (2)	C27—C26—C25	120.5 (5)
C7—P1—Os1	115.13 (13)	C27—C26—H26	119.8
C7—P1—C19	103.86 (19)	C26—C27—H27	119.1
C13—P1—Os1	115.62 (13)	C28—C27—C26	121.7 (5)
C13—P1—C7	103.79 (19)	C28—C27—H27	119.1
C13—P1—C19	103.59 (19)	C27—C28—C29	118.3 (5)
C19—P1—Os1	113.40 (14)	C27—C28—C46	121.6 (5)
C1—O1—Os1	121.4 (3)	C29—C28—C46	120.1 (5)
O1—C1—H1	115.6	C28—C29—H29	119.7
O2—C1—O1	128.8 (5)	C30—C29—C28	120.6 (5)
O2—C1—H1	115.6	C30—C29—H29	119.7
O2—Os2—Os1	83.19 (9)	C25—C30—H30	119.5
O2—Os2—P2	84.97 (9)	C29—C30—C25	121.1 (5)
O2—Os2—O4	84.76 (14)	C29—C30—H30	119.5
P2—Os2—Os1	165.90 (3)	C32—C31—P2	119.0 (3)
O4—Os2—Os1	82.05 (8)	C32—C31—C36	117.7 (4)
O4—Os2—P2	89.27 (9)	C36—C31—P2	123.3 (4)
C5—Os2—Os1	92.43 (14)	C31—C32—H32	119.2
C5—Os2—O2	95.17 (19)	C33—C32—C31	121.6 (5)
C5—Os2—P2	96.25 (14)	C33—C32—H32	119.2
C5—Os2—O4	174.46 (16)	C32—C33—H33	120.0
C6—Os2—Os1	91.97 (15)	C32—C33—C34	120.0 (5)
C6—Os2—O2	173.65 (18)	C34—C33—H33	120.0
C6—Os2—P2	99.24 (15)	C33—C34—C47	120.4 (6)
C6—Os2—O4	90.5 (2)	C35—C34—C33	119.0 (5)
C6—Os2—C5	89.1 (2)	C35—C34—C47	120.5 (6)
C1—O2—Os2	119.8 (3)	C34—C35—H35	119.3
C25—P2—Os2	116.17 (14)	C34—C35—C36	121.4 (5)
C25—P2—C37	103.6 (2)	C36—C35—H35	119.3
C31—P2—Os2	115.98 (14)	C31—C36—C35	120.1 (5)
C31—P2—C25	103.8 (2)	C31—C36—H36	119.9
C31—P2—C37	104.1 (2)	C35—C36—H36	119.9
C37—P2—Os2	111.77 (14)	C38—C37—P2	123.8 (3)
O3—C2—H2	115.8	C42—C37—P2	117.3 (3)
O4—C2—H2	115.8	C42—C37—C38	118.8 (4)
O4—C2—O3	128.4 (5)	C37—C38—H38	120.0
O5—C3—Os1	177.7 (6)	C37—C38—C39	120.0 (5)
C2—O3—Os1	120.6 (3)	C39—C38—H38	120.0
O6—C4—Os1	178.0 (5)	C38—C39—H39	119.5
C2—O4—Os2	120.7 (3)	C40—C39—C38	121.0 (5)
O7—C5—Os2	177.9 (4)	C40—C39—H39	119.5
O8—C6—Os2	177.8 (5)	C39—C40—C48	120.9 (5)
C8—C7—P1	118.3 (3)	C41—C40—C39	118.1 (4)
C12—C7—P1	123.7 (3)	C41—C40—C48	121.0 (5)
C12—C7—C8	118.0 (4)	C40—C41—H41	119.1
C7—C8—H8	119.5	C42—C41—C40	121.9 (5)
C9—C8—C7	120.9 (4)	C42—C41—H41	119.1

C9—C8—H8	119.5	C37—C42—H42	119.9
C8—C9—H9	119.5	C41—C42—C37	120.2 (4)
C10—C9—C8	120.9 (4)	C41—C42—H42	119.9
C10—C9—H9	119.5	C10—C43—H43A	109.5
C9—C10—C11	118.0 (4)	C10—C43—H43B	109.5
C9—C10—C43	120.6 (5)	C10—C43—H43C	109.5
C11—C10—C43	121.4 (5)	H43A—C43—H43B	109.5
C10—C11—H11	119.1	H43A—C43—H43C	109.5
C12—C11—C10	121.9 (5)	H43B—C43—H43C	109.5
C12—C11—H11	119.1	C16—C44—H44A	109.5
C7—C12—H12	119.9	C16—C44—H44B	109.5
C11—C12—C7	120.3 (5)	C16—C44—H44C	109.5
C11—C12—H12	119.9	H44A—C44—H44B	109.5
C14—C13—P1	123.6 (3)	H44A—C44—H44C	109.5
C18—C13—P1	118.3 (3)	H44B—C44—H44C	109.5
C18—C13—C14	118.1 (4)	C22—C45—H45A	109.5
C13—C14—H14	120.1	C22—C45—H45B	109.5
C15—C14—C13	119.9 (4)	C22—C45—H45C	109.5
C15—C14—H14	120.1	H45A—C45—H45B	109.5
C14—C15—H15	119.1	H45A—C45—H45C	109.5
C16—C15—C14	121.9 (5)	H45B—C45—H45C	109.5
C16—C15—H15	119.1	C28—C46—H46A	109.5
C15—C16—C17	117.7 (5)	C28—C46—H46B	109.5
C15—C16—C44	122.2 (5)	C28—C46—H46C	109.5
C17—C16—C44	120.1 (5)	H46A—C46—H46B	109.5
C16—C17—H17	119.1	H46A—C46—H46C	109.5
C18—C17—C16	121.8 (5)	H46B—C46—H46C	109.5
C18—C17—H17	119.1	C34—C47—H47A	109.5
C13—C18—H18	119.7	C34—C47—H47B	109.5
C17—C18—C13	120.6 (5)	C34—C47—H47C	109.5
C17—C18—H18	119.7	H47A—C47—H47B	109.5
C20—C19—P1	118.2 (3)	H47A—C47—H47C	109.5
C24—C19—P1	123.2 (3)	H47B—C47—H47C	109.5
C24—C19—C20	118.6 (4)	C40—C48—H48A	109.5
C19—C20—H20	119.4	C40—C48—H48B	109.5
C21—C20—C19	121.2 (5)	C40—C48—H48C	109.5
C21—C20—H20	119.4	H48A—C48—H48B	109.5
C20—C21—H21	119.7	H48A—C48—H48C	109.5
C20—C21—C22	120.5 (5)	H48B—C48—H48C	109.5