

Decacarbonyl(μ -ethylidenimino- $1\kappa N:2\kappa C$)- μ -hydrido-*triangulo*-triosmium(3 Os–Os)

Cynthia B. Powell,* Gregory L. Powell, Ashley K. Archambeau and Kylie M. Wilson

Department of Chemistry and Biochemistry, Abilene Christian University, ACU 28132, Abilene, Texas 79699, USA.

*Correspondence e-mail: powellc@acu.edu

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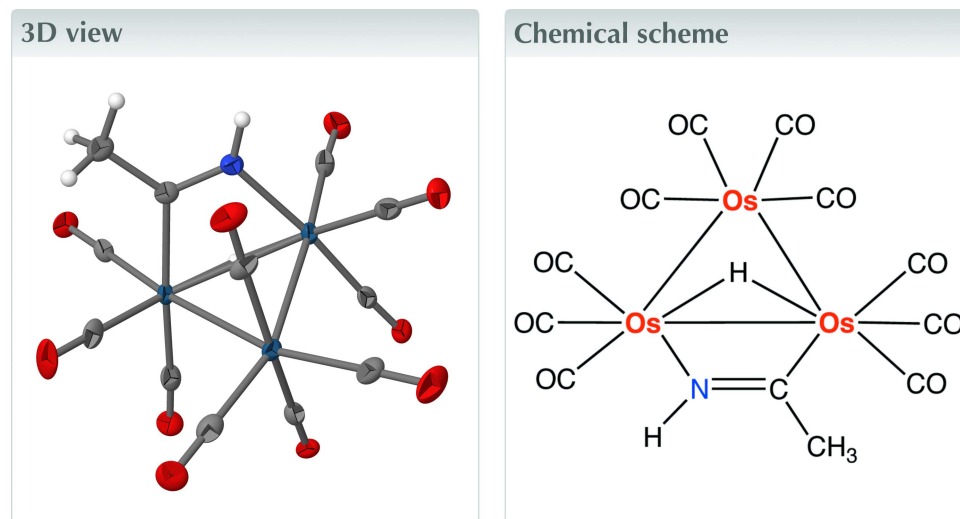
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Keywords: crystal structure; osmium trimer; triazine.

CCDC references: 1949185; 1949185

Structural data: full structural data are available from iucrdata.iucr.org

The title complex, $[\text{Os}_3(\text{C}_2\text{H}_4\text{N})\text{H}(\text{CO})_{10}]$ or $[\text{Os}_3(\text{CO})_{10}(\mu\text{-H})(\mu\text{-HN}=\text{C}-\text{CH}_3-1\kappa N:2\kappa C)]$, was synthesized in 41.6% yield by reactions between $\text{Os}_3(\text{CO})_{11}(\text{CH}_3\text{CN})$ and 2,4,6-trimethylhexahydro-1,3,5-triazine. The central osmium triangle has two Os^{I} atoms bridged by a hydride ligand and a $\mu\text{-HN}=\text{C}-\text{CH}_3-1\kappa N:2\kappa C$ triazine fragment. Three CO ligands complete the coordination sphere around each Os^{I} atom, while the remaining Os^{0} atom has four CO ligands. Each Os atom exhibits a pseudo-octahedral coordination environment, discounting the bridging Os–Os bond.



Structure description

Previous research (Liu *et al.*, 2003) has shown that 1,3,5-trimethylhexahydro-1,3,5-triazine reacts with Group 8 carbonyl compounds $M_3(\text{CO})_{12}$ ($M = \text{Fe}$ and Ru) to form $[(\mu\text{-H})M_3(\text{CO})_{11}][\text{MeN}(\text{MeNCH}_2)_2\text{CH}]$ anionic hydrido clusters, with the transfer of a hydride from the triazine to the metal carbonyl. While Fe and Ru starting materials reacted with the triazine directly, $\text{Os}_3(\text{CO})_{12}$ was first converted to $\text{Os}_3(\text{CO})_{11}(\text{CH}_3\text{CN})$ to accomplish the same result. Liu and co-workers subsequently reported the products of reactions of $\text{Os}_3(\text{CO})_{12}$ with 1,3,5-trimethylhexahydro-1,3,5-triazine, which yielded three products, *i.e.* a trimer containing a $\mu\text{-N}(\text{CH}_3)-\text{CH}_2-\text{N}(\text{CH}_3)$ triazine fragment and a hydride bridge, a trimer containing a $\mu_3\text{-N}(\text{CH}_3)-\text{CH}_2-\text{N}(\text{CH}_3)$ triazine fragment and a hydride bridge, and a dimer with two bridging $\text{N}(\text{CH}_3)-\text{CH}-\text{N}(\text{CH}_3)$ fragments perpendicular to one another forming a sawhorse-type complex (Liu *et al.*, 2005). In these complexes, each bridge has a N–C–N backbone. We were interested in further investigating the reactions of $\text{Os}_3(\text{CO})_{12}$ and $\text{Os}_3(\text{CO})_{11}(\text{CH}_3\text{CN})$ with triazines using microwave heating.

We report here the synthesis and structure of the title complex, $\text{Os}_3(\text{CO})_{10}(\mu\text{-H})(\mu\text{-HN}=\text{C}-\text{CH}_3-1\kappa N:2\kappa C)$, a trinuclear osmium compound which was the product of

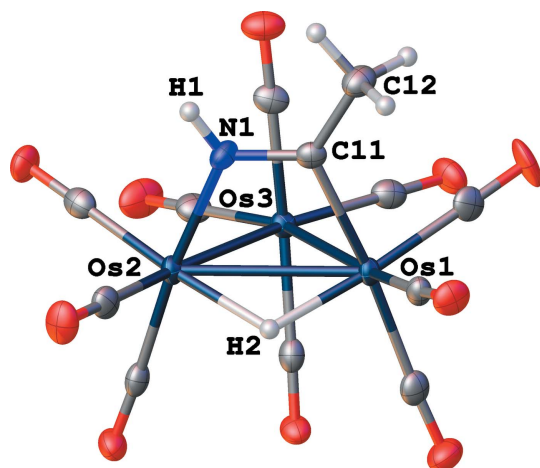


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

reactions between $\text{Os}_3(\text{CO})_{11}(\text{CH}_3\text{CN})$ and 2,4,6-trimethylhexahydro-1,3,5-triazine. The product yield was 41.6%. Rather than containing a bridge with an N–C–N backbone, $\text{Os}_3(\text{CO})_{10}(\mu\text{-H})(\mu\text{-HN}=\text{C}-\text{CH}_3\text{-}1\kappa\text{N:}2\kappa\text{C})$ contains a $\mu\text{-HN}=\text{C}-\text{CH}_3\text{-}1\kappa\text{N:}2\kappa\text{C}$ triazine fragment bridge with a N–C backbone and a hydride bridge across two Os atoms in the central osmium triangle. A few related trinuclear structures of iron, ruthenium, and osmium with bridging $\mu\text{-HN}=\text{C}-R\text{-}1\kappa\text{N:}2\kappa\text{C}$ species and nine CO ligands have been reported (Andrews *et al.*, 1978; Dawoodi *et al.*, 1981; Takao *et al.*, 2018). The only previously reported trinuclear structure which has

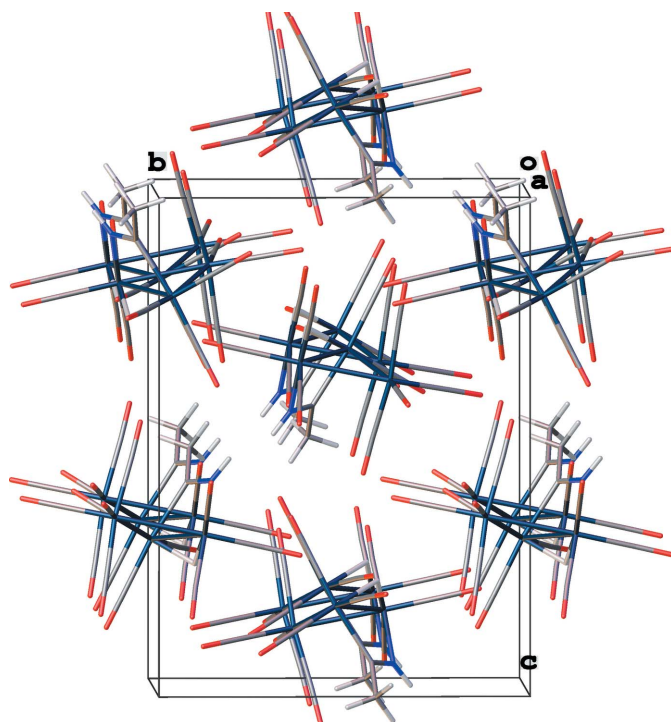


Figure 2
The packing of the molecules of the title compound in a view approximately along the *a* axis.

Table 1
Experimental details.

Crystal data	
Chemical formula	$[\text{Os}_3(\text{C}_2\text{H}_4\text{N})\text{H}(\text{CO})_{10}]$
M_r	893.77
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	100
a, b, c (Å)	9.56470 (6), 11.59555 (8), 15.61610 (11)
V (Å ³)	1731.95 (2)
Z	4
Radiation type	Cu $K\alpha$
μ (mm ⁻¹)	41.18
Crystal size (mm)	0.15 × 0.07 × 0.02
Data collection	
Diffractometer	Rigaku SuperNova AtlasS2 CCD
Absorption correction	Gaussian (<i>CrysAlis PRO</i> ; Rigaku OD, 2017)
$T_{\text{min}}, T_{\text{max}}$	0.109, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	33313, 3487, 3471
R_{int}	0.052
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.622
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.015, 0.037, 1.06
No. of reflections	3487
No. of parameters	241
No. of restraints	2
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	0.58, -0.89
Absolute structure	Refined as an inversion twin.
Absolute structure parameter	0.465 (16)

Computer programs: *CrysAlis PRO* (Rigaku OD, 2017), *SHELXL2018* (Sheldrick, 2015), *OLEX2* (Dolomanov *et al.*, 2009) and *publCIF* (Westrip, 2010).

ten CO ligands and the same $(\mu\text{-})\kappa^2\text{-N,C}(\text{H}-\text{N}=\text{C}-\text{R})$ bridging configuration as the title complex is that for $\text{Os}_3(\text{CO})_9\text{PMe}_2\text{Ph}(\mu\text{-H})(\mu\text{-HN}=\text{C}-\text{CF}_3\text{-}1\kappa\text{N:}2\kappa\text{C})$ which was synthesized in 4.3% yield by the reaction of $\text{H}_2\text{Os}_3(\text{CO})_9\text{PMe}_2\text{Ph}$ with CF_3CN (Adams *et al.*, 1981).

In the title complex, the bridged Os^1 atoms (Os1 and Os2) have three terminal CO ligands in addition to the ethylideneimino and hydride ligands, and the Os^0 atom (Os3) has four terminal CO ligands (Fig. 1). Each of the three Os atoms exhibits a pseudo-octahedral coordination environment, discounting the bridged Os–Os bond. The individual octahedra of each bridged Os atom are rotated on average by 24 (4)° out of the plane of the osmium triangle toward the mid-point of the metal–metal bond. The Os–Os bond length for the bridged bond is 2.9331 (4) Å, while the unbridged Os–Os bond lengths are shorter, as expected, at 2.8604 (4) and 2.8759 (4) Å. The molecules stack so that the planes containing the triangular Os_3 units are roughly perpendicular to the *c* axis (Fig. 2). A 2_1 screw axis passes through the centers of the Os_3 triangles. Thus, every Os_3 unit in the stack is rotated 180° from the one above it and below it so that the triazine fragments of every other molecule are facing in the opposite direction. Although an N–H donor group is present, there is no evidence of classical hydrogen bonding in the crystal.

Synthesis and crystallization

Dodecacarbonyltriosmium(0) (60.2 mg, 0.066 mmol) and CH₃CN (7.5 ml) were placed in a 35 ml glass reaction vessel, then sealed with a PTFE cap and placed in a CEM Discover-SP microwave reactor. The mixture was stirred and heated at 411 K for 9 min to yield a green solution of Os₃(CO)₁₁(CH₃CN) (Jung *et al.*, 2009). The reaction vessel was removed from the microwave reactor. The solvent was removed by rotary evaporation. 1,2-Dichloroethane (7 ml) was added to the dry Os₃(CO)₁₁(CH₃CN). Acetaldehyde ammonia trimer (61.2 mg, 0.474 mmol) was then added to the vessel, which was then sealed with a PTFE cap, and the mixture was stirred and heated in a microwave reactor at 398 K for 20 min to produce a yellow–orange solution. The solvent was then removed by rotary evaporation, and the residue was dissolved in CH₂Cl₂ and subjected to thin-layer chromatography using an eluent mixture of 2.5:1 (v/v) hexanes/CH₂Cl₂. Two yellow bands were collected. The top band contained 2.1 mg of an unidentified compound and had an *R_F* value of 0.82. IR (νCO, CHCl₃): 2103 (*w*), 2065 (*vs*), 2050 (*m*), 2034 (*w*), 2017 (*s*), 2001 (*m*), 1988 (*sh*) cm⁻¹. The second band consisted of 24.7 mg of the title complex (41.6% yield) and had an *R_F* value of 0.67. IR (νCO, *n*-hexane): 2105 (*w*), 2064 (*vs*), 2052 (*s*), 2024 (*s*), 2007 (*vs*), 1991 (*m*), 1977 (*w*) cm⁻¹. ¹H NMR (60 MHz, CDCl₃): δ 2.18 (*s*, 3H, CH₃), -15.15 (*s*, 1H, Os–H–Os). Crystals were grown at 277 K *via p*-xylene vapor diffusion into a CHCl₃ solution.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. The crystal under investigation was twinned by inversion; TWIN and BASF commands were used to refine the absolute structure parameter for this noncen-

trosymmetric structure. The bridging hydride ligand and the N-bound H atom were both located in a difference Fourier map. A DFIX command was used to constrain the position of the hydride H atom, while an AFIX command was used to constrain the N-bound H atom.

Acknowledgements

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full crystallographic data

IUCrData (2019), 4, x191386 [https://doi.org/10.1107/S2414314619013865]

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Decacarbonyl(μ -ethylidenimino- $1\kappa N:2\kappa C$)- μ -hydrido-triangulo-triosmium(3 Os–Os)

Crystal data

[Os₃(C₂H₄N)H(CO)₁₀]

$M_r = 893.77$

Orthorhombic, $P2_12_12_1$

$a = 9.56470$ (6) Å

$b = 11.59555$ (8) Å

$c = 15.61610$ (11) Å

$V = 1731.95$ (2) Å³

$Z = 4$

$F(000) = 1568$

$D_x = 3.428$ Mg m⁻³

Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å

Cell parameters from 23076 reflections

$\theta = 3.8$ – 73.5°

$\mu = 41.18$ mm⁻¹

$T = 100$ K

Plate, clear reddish orange

$0.15 \times 0.07 \times 0.02$ 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)

$T_{\min} = 0.109$, $T_{\max} = 1.000$

33313 measured reflections

3487 independent reflections

3471 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.052$

$\theta_{\max} = 73.6^\circ$, $\theta_{\min} = 4.8^\circ$

$h = -11 \rightarrow 11$

$k = -14 \rightarrow 14$

$l = -19 \rightarrow 19$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.015$

$wR(F^2) = 0.037$

$S = 1.06$

3487 reflections

241 parameters

2 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0177P)^2 + 1.930P]$

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

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.58$ e Å⁻³

$\Delta\rho_{\min} = -0.89$ e Å⁻³

Absolute structure: Refined as an inversion
twin.

Absolute structure parameter: 0.465 (16)

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.

Refinement. An AFIX command was used to constrain the N-bound H atom. A DFIX command was used to constrain the position of hydride H atom.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Os1	0.55519 (3)	0.47729 (2)	0.31968 (2)	0.01492 (7)
Os3	0.81319 (3)	0.37203 (2)	0.37293 (2)	0.01544 (7)
Os2	0.81157 (3)	0.61402 (2)	0.33737 (2)	0.01578 (7)
O8	0.7118 (6)	0.1256 (5)	0.4066 (3)	0.0295 (12)
O2	0.2816 (6)	0.6049 (5)	0.2863 (3)	0.0265 (11)
N1	0.6712 (6)	0.6362 (5)	0.4404 (3)	0.0192 (11)
H1	0.687362	0.682623	0.483836	0.023*
O6	1.0711 (6)	0.6239 (5)	0.4504 (3)	0.0270 (11)
O5	0.7938 (6)	0.8712 (4)	0.2948 (4)	0.0309 (12)
O1	0.5513 (7)	0.3569 (5)	0.1421 (3)	0.0317 (12)
O7	0.8601 (5)	0.3331 (4)	0.1797 (3)	0.0218 (10)
O9	1.1229 (6)	0.3324 (6)	0.4109 (4)	0.0375 (15)
O3	0.4224 (6)	0.2725 (5)	0.4103 (4)	0.0355 (13)
O4	0.9924 (5)	0.5754 (5)	0.1790 (3)	0.0246 (11)
C8	0.7491 (7)	0.2157 (7)	0.3941 (4)	0.0237 (15)
C1	0.5576 (8)	0.4015 (6)	0.2072 (4)	0.0235 (14)
O10	0.7542 (6)	0.4424 (5)	0.5608 (3)	0.0292 (12)
C11	0.5588 (8)	0.5746 (6)	0.4332 (4)	0.0179 (13)
C9	1.0074 (8)	0.3481 (7)	0.3968 (5)	0.0254 (16)
C6	0.9740 (8)	0.6201 (6)	0.4085 (5)	0.0225 (15)
C3	0.4709 (7)	0.3493 (7)	0.3761 (5)	0.0238 (16)
C4	0.9231 (7)	0.5866 (6)	0.2380 (4)	0.0199 (14)
C10	0.7726 (7)	0.4187 (6)	0.4916 (4)	0.0209 (15)
C12	0.4450 (8)	0.5834 (7)	0.4983 (4)	0.0259 (15)
H12A	0.363183	0.620690	0.472632	0.039*
H12B	0.477705	0.629330	0.547069	0.039*
H12C	0.419399	0.506005	0.518069	0.039*
C5	0.7956 (7)	0.7757 (6)	0.3109 (5)	0.0231 (14)
C7	0.8407 (7)	0.3485 (6)	0.2497 (5)	0.0206 (14)
C2	0.3870 (7)	0.5583 (6)	0.2960 (4)	0.0190 (14)
H2	0.652 (9)	0.592 (9)	0.273 (6)	0.09 (5)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Os1	0.01033 (13)	0.01544 (13)	0.01901 (13)	−0.00051 (10)	−0.00092 (12)	0.00164 (10)
Os3	0.01224 (14)	0.01589 (12)	0.01820 (13)	0.00247 (12)	0.00051 (11)	0.00095 (10)
Os2	0.01117 (13)	0.01577 (13)	0.02039 (13)	−0.00120 (11)	−0.00046 (11)	0.00136 (10)
O8	0.037 (3)	0.019 (2)	0.033 (3)	−0.003 (2)	0.008 (2)	0.005 (2)
O2	0.021 (3)	0.030 (3)	0.029 (2)	0.009 (2)	−0.007 (2)	0.001 (2)
N1	0.017 (3)	0.021 (3)	0.020 (3)	0.002 (3)	−0.004 (2)	−0.005 (2)
O6	0.016 (2)	0.033 (3)	0.032 (3)	−0.001 (2)	−0.007 (2)	−0.002 (2)

O5	0.031 (3)	0.018 (2)	0.044 (3)	0.000 (2)	-0.007 (2)	0.007 (2)
O1	0.031 (3)	0.039 (3)	0.025 (3)	-0.003 (3)	-0.002 (2)	-0.010 (2)
O7	0.024 (3)	0.020 (2)	0.021 (3)	0.0001 (19)	0.006 (2)	-0.005 (2)
O9	0.019 (3)	0.057 (4)	0.037 (3)	0.015 (3)	-0.005 (2)	-0.006 (3)
O3	0.022 (3)	0.027 (3)	0.057 (4)	-0.007 (2)	0.009 (3)	0.022 (3)
O4	0.020 (2)	0.028 (3)	0.026 (3)	-0.004 (2)	0.005 (2)	-0.003 (2)
C8	0.021 (4)	0.029 (4)	0.021 (3)	0.006 (3)	0.001 (3)	0.001 (3)
C1	0.015 (3)	0.026 (4)	0.029 (3)	0.001 (3)	-0.005 (3)	0.003 (3)
O10	0.035 (3)	0.033 (3)	0.020 (3)	0.013 (2)	0.002 (2)	0.000 (2)
C11	0.020 (3)	0.017 (3)	0.017 (3)	0.004 (3)	-0.002 (3)	0.002 (2)
C9	0.024 (4)	0.030 (4)	0.022 (3)	0.004 (3)	0.006 (3)	-0.003 (3)
C6	0.028 (4)	0.016 (3)	0.024 (3)	0.001 (3)	0.005 (3)	0.000 (3)
C3	0.011 (3)	0.032 (4)	0.029 (3)	0.005 (3)	-0.004 (3)	0.002 (3)
C4	0.014 (3)	0.018 (3)	0.028 (3)	0.000 (3)	-0.006 (3)	-0.001 (3)
C10	0.021 (4)	0.021 (3)	0.021 (4)	0.010 (3)	0.002 (3)	0.003 (3)
C12	0.023 (4)	0.031 (4)	0.024 (3)	0.003 (3)	0.000 (3)	-0.006 (3)
C5	0.014 (3)	0.029 (4)	0.025 (3)	-0.001 (3)	-0.004 (3)	0.003 (3)
C7	0.016 (3)	0.017 (3)	0.029 (4)	-0.001 (2)	-0.001 (3)	0.000 (3)
C2	0.018 (4)	0.019 (3)	0.020 (3)	-0.003 (3)	-0.003 (3)	0.003 (3)

Geometric parameters (Å, °)

Os1—Os3	2.8759 (4)	O8—C8	1.121 (10)
Os1—Os2	2.9331 (4)	O2—C2	1.154 (9)
Os1—C1	1.964 (7)	N1—H1	0.8800
Os1—C11	2.102 (6)	N1—C11	1.296 (10)
Os1—C3	1.904 (8)	O6—C6	1.137 (9)
Os1—C2	1.899 (7)	O5—C5	1.136 (9)
Os1—H2	1.78 (6)	O1—C1	1.142 (9)
Os3—Os2	2.8604 (4)	O7—C7	1.124 (9)
Os3—C8	1.942 (8)	O9—C9	1.142 (10)
Os3—C9	1.914 (8)	O3—C3	1.138 (9)
Os3—C10	1.969 (7)	O4—C4	1.141 (9)
Os3—C7	1.961 (7)	O10—C10	1.128 (9)
Os2—N1	2.111 (6)	C11—C12	1.493 (10)
Os2—C6	1.911 (7)	C12—H12A	0.9800
Os2—C4	1.910 (7)	C12—H12B	0.9800
Os2—C5	1.926 (7)	C12—H12C	0.9800
Os2—H2	1.84 (6)		
Os3—Os1—Os2	58.988 (9)	N1—Os2—Os3	88.56 (16)
Os3—Os1—H2	89 (3)	N1—Os2—H2	85 (4)
Os2—Os1—H2	37 (2)	C6—Os2—Os1	139.0 (2)
C1—Os1—Os3	93.4 (2)	C6—Os2—Os3	85.3 (2)
C1—Os1—Os2	108.4 (2)	C6—Os2—N1	94.0 (3)
C1—Os1—C11	173.9 (3)	C6—Os2—C5	98.8 (3)
C1—Os1—H2	88 (4)	C6—Os2—H2	174 (2)
C11—Os1—Os3	88.3 (2)	C4—Os2—Os1	107.5 (2)

C11—Os1—Os2	67.5 (2)	C4—Os2—Os3	89.5 (2)
C11—Os1—H2	86 (4)	C4—Os2—N1	174.1 (3)
C3—Os1—Os3	84.2 (2)	C4—Os2—C6	91.4 (3)
C3—Os1—Os2	137.0 (2)	C4—Os2—C5	91.8 (3)
C3—Os1—C1	94.0 (3)	C4—Os2—H2	90 (4)
C3—Os1—C11	92.0 (3)	C5—Os2—Os1	116.1 (2)
C3—Os1—H2	173 (2)	C5—Os2—Os3	175.6 (2)
C2—Os1—Os3	173.2 (2)	C5—Os2—N1	89.7 (3)
C2—Os1—Os2	117.3 (2)	C5—Os2—H2	87 (3)
C2—Os1—C1	93.3 (3)	Os2—N1—H1	123.4
C2—Os1—C11	85.0 (3)	C11—N1—Os2	113.3 (4)
C2—Os1—C3	96.7 (3)	C11—N1—H1	123.4
C2—Os1—H2	90 (3)	O8—C8—Os3	179.7 (7)
Os2—Os3—Os1	61.502 (10)	O1—C1—Os1	176.3 (7)
C8—Os3—Os1	100.1 (2)	N1—C11—Os1	112.5 (5)
C8—Os3—Os2	161.3 (2)	N1—C11—C12	120.6 (6)
C8—Os3—C10	92.0 (3)	C12—C11—Os1	126.8 (5)
C8—Os3—C7	94.6 (3)	O9—C9—Os3	179.2 (8)
C9—Os3—Os1	161.8 (2)	O6—C6—Os2	179.6 (7)
C9—Os3—Os2	100.7 (2)	O3—C3—Os1	179.0 (7)
C9—Os3—C8	97.9 (3)	O4—C4—Os2	176.7 (6)
C9—Os3—C10	92.8 (3)	O10—C10—Os3	176.9 (6)
C9—Os3—C7	92.3 (3)	C11—C12—H12A	109.5
C10—Os3—Os1	89.2 (2)	C11—C12—H12B	109.5
C10—Os3—Os2	85.0 (2)	C11—C12—H12C	109.5
C7—Os3—Os1	83.7 (2)	H12A—C12—H12B	109.5
C7—Os3—Os2	86.9 (2)	H12A—C12—H12C	109.5
C7—Os3—C10	171.1 (3)	H12B—C12—H12C	109.5
Os1—Os2—H2	35 (2)	O5—C5—Os2	176.3 (7)
Os3—Os2—Os1	59.509 (9)	O7—C7—Os3	177.8 (6)
Os3—Os2—H2	88 (3)	O2—C2—Os1	175.8 (6)
N1—Os2—Os1	66.78 (15)		
Os2—N1—C11—Os1	-1.3 (6)	Os2—N1—C11—C12	-177.2 (5)
