

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

	$U_{\text{eq}} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$
Os(1)	0.58045 (2)
Os(2)	0.58275 (2)
Ge(1)	0.62469 (4)
O(11)	0.5489 (4)
O(12)	0.5988 (4)
O(13)	0.6570 (4)
O(14)	0.5206 (4)
O(21)	0.6014 (4)
O(22)	0.5519 (4)
O(23)	0.6611 (4)
O(24)	0.5225 (4)
C(11)	0.5606 (4)
C(12)	0.5933 (5)
C(13)	0.6295 (5)
C(14)	0.5422 (5)
C(21)	0.5956 (5)
C(22)	0.5642 (5)
C(23)	0.6324 (5)
C(24)	0.5453 (6)
C(101)	0.6585 (2)
C(102)	0.6864 (3)
C(103)	0.7129 (2)
C(104)	0.7115 (3)
C(105)	0.6836 (3)
C(106)	0.6571 (2)
C(201)	0.6684 (2)
C(202)	0.6625 (2)
C(203)	0.6941 (3)
C(204)	0.7317 (2)
C(205)	0.7376 (2)
C(206)	0.7060 (3)
x	0.04319 (6)
y	0.43598 (7)
z	0.16885 (4)
U_{eq}	0.0628
	0.0736
	0.0584
	0.1091
	0.0947
	0.0949
	0.1070
	0.1203
	0.1121
	0.1136
	0.1211
	0.0782
	0.0711
	0.0708
	0.0852
	0.0825
	0.0897
	0.0857
	0.0944
	0.0584
	0.0626
	0.0657
	0.0626
	0.0600
	0.0627
	0.0679
	0.0935
	0.0679
	0.0627

Table 2. Selected geometric parameters (\AA , $^\circ$)

Os(1)—Ge(1)	2.599 (2)	Ge(1)—C(101)	1.992 (6)
Os(1)—C(11)	1.95 (2)	Ge(1)—C(201)	1.986 (6)
Os(1)—C(12)	1.94 (2)	O(11)—C(11)	1.13 (2)
Os(1)—C(13)	2.00 (2)	O(12)—C(12)	1.13 (1)
Os(1)—C(14)	1.98 (2)	O(13)—C(13)	1.11 (2)
Os(2)—Ge(1)	2.614 (2)	O(14)—C(14)	1.10 (2)
Os(2)—C(21)	1.97 (2)	O(21)—C(21)	1.11 (2)
Os(2)—C(22)	1.93 (2)	O(22)—C(22)	1.16 (2)
Os(2)—C(23)	1.99 (2)	O(23)—C(23)	1.14 (2)
Os(2)—C(24)	1.92 (2)	O(24)—C(24)	1.16 (2)
Os(1)—Ge(1)—Os(2)	114.41 (6)	Os(2)—C(21)—O(21)	177.5 (16)
Os(1)—C(11)—O(11)	170.2 (17)	Os(2)—C(22)—O(22)	176.1 (18)
Os(1)—C(12)—O(12)	174.9 (14)	Os(2)—C(23)—O(23)	178.2 (17)
Os(1)—C(13)—O(13)	178.7 (15)	Os(2)—C(24)—O(24)	178.5 (19)
Os(1)—C(14)—O(14)	178.8 (16)		

The phenyl C atoms were regularized, with C—C bond lengths of 1.39 \AA , and the phenyl H atoms were placed in calculated positions, with C—H bond lengths of 1.0 \AA . The phenyl rings were refined as rigid groups pivoted about dummy atoms placed at the centroids. Equivalent phenyl C atoms were given the same displacement parameters and their shifts made equivalent, giving four sets of U_{ij} per phenyl ring. The metal hydrides were also placed in calculated positions, at 1.66 \AA from their attached Os atoms and *trans* to a CO group.

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *NRCVAX DATRD2* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *NRCVAX SOLVER*. Program(s) used to refine structure: *CRYSTALS* (Watkin, Carruthers & Betteridge, 1985). Molecular graphics: *NRCVAX*. Software used to prepare material for publication: *NRCVAX TABLES*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1150). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Two Chloro-Bridged Osmium Clusters

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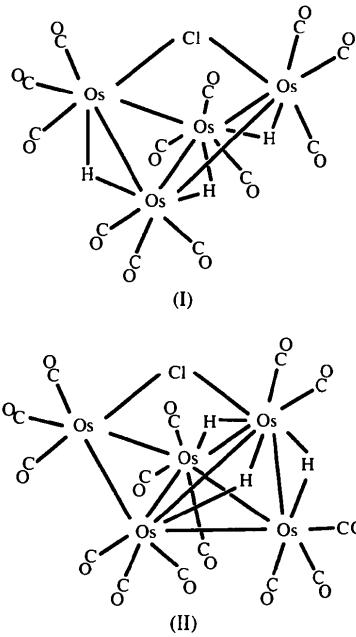
Abstract

The molecular structure of the chloro-bridged tetraosmium cluster, dodecacarbonyl-1 κ^3 C,2 κ^3 C,3 κ^3 C,4 κ^3 C- μ -chloro-3:4 κ^2 Cl-tris- μ -hydrido-1:2 κ^2 H;1:3 κ^2 H;2:4 κ^2 H-tetraosmium(5 Os—Os), [Os₄(μ -Cl)(μ -H)₃(CO)₁₂], consists of a ‘butterfly’ arrangement of Os atoms, just as in the iodo analogue. The chloro bridge in the tetraosmium cluster is symmetric, whereas in the pentaosmium cluster, tetradecacarbonyl-1 κ^3 C,2 κ^2 C,3 κ^3 C,4 κ^3 C,5 κ^3 - μ -chloro-2:5 κ^2 Cl-tris- μ -hydrido-1:2 κ^2 H;2:3 κ^2 H;2:4 κ^2 H-

pentaosmium(8 Os—Os), $[Os_5(\mu\text{-Cl})(\mu\text{-H})_3(CO)_{14}]$, it is distinctly asymmetric. This asymmetry is attributed to the *trans* influence of a carbonyl ligand, which lengthens the *trans*-Os—Cl bond.

Comment

The two title chloro-bridged clusters $[Os_4(\mu\text{-Cl})(\mu\text{-H})_3(CO)_{12}]$, (I), and $[Os_5(\mu\text{-Cl})(\mu\text{-H})_3(CO)_{14}]$, (II), were obtained in low yield from the reaction of impure $MeSnH_3$ with $[Os_3(\mu\text{-H})_2(CO)_{10}]$ at 353 K. The impurities are believed to be $MeSnH_{3-x}Cl_x$ ($x = 0\text{--}2$) arising from incomplete reduction of $MeSnCl_3$ by $LiAlH_4$ used in the preparation of $MeSnH_3$.



In both clusters, the metal hydrides were placed in calculated positions (Orpen, 1980). Although not iso-morphous, the tetraosmium cluster [(I); Fig. 1] has the same ‘butterfly’ structure as the known iodo analogue $[Os_4(\mu\text{-I})(\mu\text{-H})_3(CO)_{12}]$; the structure of the iodo compound was determined by both X-ray and neutron-diffraction methods (Johnson, Lewis, Raithby, Wong & Rouse, 1980). As expected, the smaller size of the Cl atom leads to a larger Os—X—Os ($X = \text{halogen}$) angle and shorter Os—X bond lengths. Both tetraosmium clusters show the same lengthening of the hydride-bridged Os—Os bonds compared with the unbridged Os—Os bonds; in the chloro compound, these range from 2.935 (1) to 3.0114 (9) Å and from 2.848 (1) to 2.849 (1) Å, respectively, while in the iodo analogue, they are 2.927 (2) and 3.055 (1) Å, and 2.877 Å, respectively. Furthermore, the shortest hydride-bridged Os—Os bond in each cluster is the ‘hinge’ bond, *viz.* the Os₂—Os₄ bond in the cluster reported here. It is also noteworthy that the assumed positions of the hydride

ligands imply a chirality of both the chloro and iodo tetraosmium clusters, *i.e.* each crystal may be regarded as comprising a racemic mixture, although it seems likely that the hydride ligands are fluxional in solution. The tetraosmium cluster also possesses an approximate molecular non-crystallographic twofold axis through the Cl atom and the midpoint of the ‘hinge’ bond [detected by the *BUNYIP* program (Hester & Hall, 1996)].

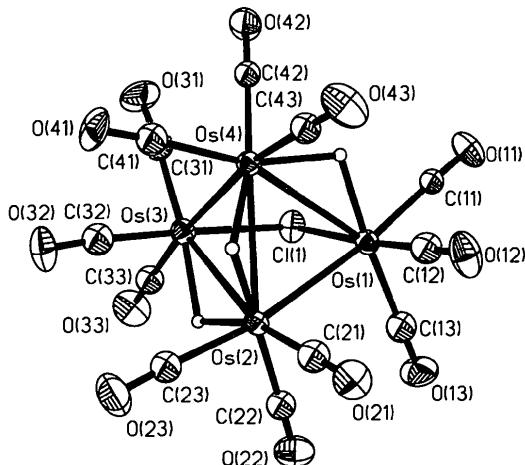


Fig. 1. ORTEPII (Johnson, 1976) diagram (30% probability displacement ellipsoids) showing the atomic numbering scheme for $[Os_4(\mu\text{-Cl})(\mu\text{-H})_3(CO)_{12}]$.

The osmium skeleton of the pentaosmium cluster [(II); Fig. 2] is the same as that of the pentaosmium hydridocarbonyl cluster $[Os_5(\mu\text{-H})_2(CO)_{16}]$ (Guy & Sheldrick, 1978). There is also structural similarity between $[Os_4(\mu\text{-Cl})(\mu\text{-H})_3(CO)_{12}]$ (described above) and $[Os_5(\mu\text{-Cl})(\mu\text{-H})_3(CO)_{14}]$ in that the latter can be regarded as derived from the former by capping one ‘wing’ of the ‘butterfly’; the location of the hydrides

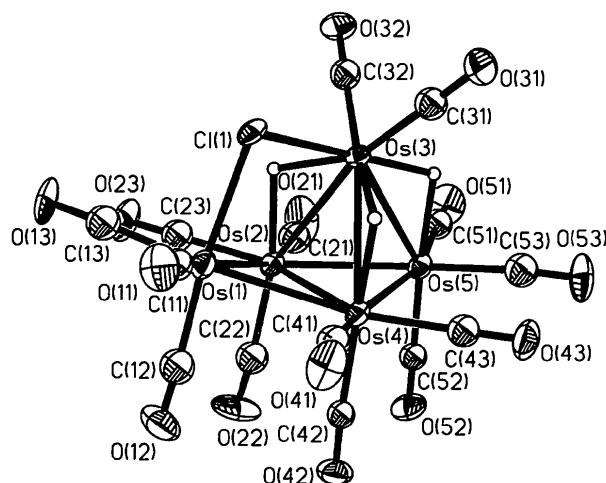


Fig. 2. ORTEPII (Johnson, 1976) diagram (30% probability displacement ellipsoids) showing the atomic numbering scheme for $[Os_5(\mu\text{-Cl})(\mu\text{-H})_3(CO)_{14}]$.

in the two clusters are, however, quite different. Although the hydride-bridged Os—Os bonds [2.895(1)–2.945(1) Å] are again lengthened compared with the unbridged Os—Os bonds [2.828(1)–2.875(1) Å], this effect appears to be less than in the more open tetraosmium cluster. One of the most significant structural differences between the tetraosmium and pentaosmium clusters is that the chloro bridge in the latter is distinctly asymmetric; the Os—Cl bonds are 2.407(6) and 2.478(6) Å, respectively. This may be attributed to the absence of a carbonyl *trans* to the chloro bridge on the Os₃ atom; presumably, the presence of a *trans* carbonyl on Os₁ decreases the Os to Cl *dπ–pπ* bonding, hence lengthening the Os₁—Cl₁ bond compared with the Os₃—Cl₁ bond.

Experimental

MeSnCl₃ and excess LiAlH₄ were placed in a Carius tube, which was then evacuated and diethyl ether vacuum transferred in. The reaction mixture was stirred at 263 K for 45 min and then the volatiles were vacuum transferred into a previously evacuated Carius tube containing [Os₃(μ-H)₂(CO)₁₀] (0.33 equivalents). The resulting mixture was stirred at room temperature overnight, then heated at 353 K for 3 d. The solvent and volatiles were then removed *in vacuo* and the residue extracted with warm hexane followed by column chromatography on silica. Elution with hexane gave a purple band of the starting cluster, followed by a lemon yellow band, which upon concentration and cooling gave yellow crystals of [Os₄(μ-Cl)(μ-H)₃(CO)₁₂] and [Os₅(μ-Cl)(μ-H)₃(CO)₁₄].

Compound (I)

Crystal data

[Os₄Cl(H)₃(CO)₁₂]

$M_r = 1135.40$

Monoclinic

$P2_1/n$

$a = 9.5247(8)$ Å

$b = 16.7749(18)$ Å

$c = 12.9718(19)$ Å

$\beta = 97.574(9)^\circ$

$V = 2054.5(4)$ Å³

$Z = 4$

$D_x = 3.671$ Mg m⁻³

D_m not measured

Data collection

Nonius CAD-4 diffractometer

$w/2\theta$ scans

Absorption correction:

ψ scan and spherical (North, Phillips & Matthews, 1968)

$T_{\min} = 0.0139$, $T_{\max} = 0.0487$

2940 measured reflections

2842 independent reflections

Mo $K\alpha$ radiation

$\lambda = 0.71069$ Å
Cell parameters from 25

reflections

$\theta = 15.5$ –20.0°

$\mu = 24.88$ mm⁻¹

$T = 294$ K

Hexagonal prism

0.42 × 0.33 × 0.24 mm

Yellow

Refinement

Refinement on F

$R = 0.038$

$wR = 0.041$

$S = 3.2$

2453 reflections

203 parameters

$$w = 1/[\sigma^2(F_o) + 0.00005|F_o|^2]$$

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

$$\Delta\rho_{\text{max}} = 2.4(3) \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\text{min}} = -1.6(3) \text{ e } \text{\AA}^{-3}$$

Extinction correction:

Larson (1970)

Extinction coefficient:

0.058 (10)

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²) for (I)

	x	y	z	U_{eq}
Os1	0.71691(7)	0.84954(4)	0.06846(5)	0.036
Os2	1.00660(7)	0.80307(4)	0.10918(5)	0.035
Os3	0.86445(8)	0.65701(4)	0.00956(5)	0.040
Os4	0.80075(7)	0.70887(4)	0.20724(5)	0.036
C11	0.6771(5)	0.7483(3)	−0.0706(4)	0.0496
O11	0.4040(15)	0.8842(9)	0.0393(11)	0.0655
O12	0.7685(16)	0.9621(9)	0.2534(12)	0.0670
O13	0.7892(17)	0.9702(9)	−0.0961(12)	0.0736
O21	1.0900(16)	0.9357(8)	0.2621(11)	0.0663
O22	1.0867(18)	0.9001(10)	−0.0689(12)	0.0778
O23	1.2985(17)	0.7291(10)	0.1670(14)	0.0844
O31	0.6574(18)	0.5150(9)	0.0091(13)	0.0767
O32	1.1061(17)	0.5587(8)	0.1096(13)	0.0732
O33	0.9456(18)	0.6213(10)	−0.2042(11)	0.0786
O41	0.9749(16)	0.5741(8)	0.3151(12)	0.0584
O42	0.5360(14)	0.6077(8)	0.1856(12)	0.0627
O43	0.7610(19)	0.7913(10)	0.4120(11)	0.0757
C11	0.516(2)	0.8703(10)	0.0495(13)	0.038(4)
C12	0.747(2)	0.9189(12)	0.1841(16)	0.055(5)
C13	0.765(2)	0.9277(12)	−0.0371(16)	0.059(5)
C21	1.058(2)	0.8848(12)	0.2057(16)	0.054(5)
C22	1.057(2)	0.8644(11)	−0.0028(15)	0.050(5)
C23	1.191(2)	0.7540(11)	0.1436(15)	0.051(5)
C31	0.731(2)	0.5700(12)	0.0098(16)	0.059(5)
C32	1.012(2)	0.5946(13)	0.0762(16)	0.061(5)
C33	0.914(2)	0.6337(11)	−0.1235(16)	0.055(5)
C41	0.907(2)	0.6210(12)	0.2749(15)	0.054(5)
C42	0.631(2)	0.6454(10)	0.1919(13)	0.041(4)
C43	0.777(2)	0.7591(11)	0.3358(15)	0.047(5)

Table 2. Selected geometric parameters (Å, °) for (I)

Os1—Os2	2.849(1)	Os3—Os4	2.848(1)
Os1—Os4	3.0114(9)	Os1—C11	2.471(4)
Os2—Os3	3.007(1)	Os3—C11	2.474(5)
Os2—Os4	2.935(1)		
Os2—Os1—Os4	60.04(2)	Os1—Os4—Os2	57.23(2)
Os1—Os2—Os3	76.89(3)	Os1—Os4—Os3	76.83(3)
Os1—Os2—Os4	62.73(2)	Os2—Os4—Os3	62.64(2)
Os3—Os2—Os4	57.25(2)	Os1—C11—Os3	94.9(2)
Os2—Os3—Os4	60.10(2)		

Compound (II)

Crystal data

[Os₅Cl(H)₃(CO)₁₄]
 $M_r = 1381.62$

Mo $K\alpha$ radiation

$\lambda = 0.71069$ Å

Triclinic	Cell parameters from 25
<i>P</i> 1	reflections
<i>a</i> = 8.8621 (15) Å	θ = 15.00–18.15°
<i>b</i> = 9.3825 (15) Å	μ = 27.45 mm ⁻¹
<i>c</i> = 15.9148 (23) Å	<i>T</i> = 294 K
α = 80.815 (13)°	Irregular plate
β = 85.332 (13)°	0.19 × 0.16 × 0.08 mm
γ = 62.828 (13)°	Red
<i>V</i> = 1162.1 (3) Å ³	
<i>Z</i> = 2	
D_x = 3.948 Mg m ⁻³	
D_m not measured	

Data collection

Nonius CAD-4 diffractometer

 $\omega/2\theta$ scans

Absorption correction:

Gaussian by integration
from crystal shape
(Busing & Levy, 1957) T_{\min} = 0.0273, T_{\max} = 0.16873274 measured reflections
3012 independent reflections*Refinement*Refinement on *F* R = 0.054 wR = 0.063 S = 3.2

2536 reflections

237 parameters

 $w = 1/[\sigma^2(F_o) + 0.0002|F_o|^2]$
 $(\Delta/\sigma)_{\max} = 0.04$

2536 observed reflections

[$I_{\text{net}} > 2.5\sigma(I_{\text{net}})$] R_{int} = 0.050 θ_{\max} = 22.5° h = -8 → 9 k = 0 → 10 l = -16 → 17

2 standard reflections

frequency: 60 min

intensity decay: 2.0%

 $\Delta\rho_{\max} = 2.7$ (5) e Å⁻³
 $\Delta\rho_{\min} = -3.7$ (5) e Å⁻³
Extinction correction: none
Atomic scattering factors
from *International Tables*
for X-ray Crystallography
(1974, Vol. IV, Table
2.2B)
Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²) for (II)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
Os1	0.80842 (13)	0.27859 (12)	0.14057 (7)	0.03932
Os2	0.92928 (12)	0.34997 (11)	0.28145 (6)	0.03527
Os3	0.76671 (12)	0.66189 (11)	0.17241 (6)	0.03418
Os4	0.57629 (12)	0.48878 (11)	0.25493 (6)	0.03231
Os5	0.70828 (12)	0.62988 (12)	0.35458 (6)	0.03578
C11	0.8173 (8)	0.5178 (8)	0.0526 (4)	0.0395
O11	0.602 (3)	0.274 (3)	-0.0009 (14)	0.0706
O12	0.785 (4)	0.009 (3)	0.2590 (15)	0.0752
O13	1.131 (3)	0.059 (3)	0.0511 (15)	0.0632
O21	1.166 (3)	0.414 (3)	0.385 (2)	0.0722
O22	0.933 (3)	0.083 (3)	0.4180 (17)	0.0729
O23	1.257 (3)	0.107 (3)	0.2108 (16)	0.0704
O31	0.520 (3)	0.992 (2)	0.0853 (15)	0.0723
O32	1.044 (2)	0.765 (2)	0.1318 (14)	0.0581
O41	0.349 (3)	0.432 (3)	0.1435 (15)	0.0701
O42	0.539 (3)	0.251 (2)	0.3971 (15)	0.0583
O43	0.247 (2)	0.769 (2)	0.3010 (15)	0.0629
O51	0.933 (3)	0.738 (3)	0.4393 (16)	0.0692
O52	0.690 (3)	0.416 (2)	0.5156 (12)	0.0584
O53	0.391 (3)	0.930 (2)	0.3985 (14)	0.0627
C11	0.675 (3)	0.279 (3)	0.0478 (19)	0.047 (7)
C12	0.792 (4)	0.108 (4)	0.218 (2)	0.064 (9)
C13	1.019 (4)	0.139 (4)	0.085 (2)	0.064 (8)
C21	1.072 (4)	0.394 (4)	0.350 (2)	0.068 (9)
C22	0.923 (4)	0.185 (4)	0.369 (2)	0.064 (8)
C23	1.131 (4)	0.197 (4)	0.232 (2)	0.060 (8)

C31	0.615 (4)	0.862 (4)	0.1195 (19)	0.055 (7)
C32	0.942 (4)	0.725 (3)	0.1485 (19)	0.054 (7)
C41	0.441 (4)	0.448 (3)	0.180 (2)	0.054 (7)
C42	0.558 (3)	0.334 (3)	0.3453 (18)	0.042 (6)
C43	0.373 (4)	0.665 (3)	0.2885 (19)	0.054 (7)
C51	0.850 (4)	0.701 (3)	0.405 (2)	0.059 (8)
C52	0.696 (3)	0.495 (3)	0.457 (2)	0.046 (7)
C53	0.508 (4)	0.824 (4)	0.378 (2)	0.067 (9)

Table 4. Selected geometric parameters (Å, °) for (II)

Os1—Os2	2.855 (2)	Os3—Os4	2.945 (1)
Os1—Os4	2.875 (1)	Os3—Os5	2.895 (1)
Os2—Os3	2.930 (1)	Os4—Os5	2.832 (1)
Os2—Os4	2.825 (1)	Os1—C11	2.478 (6)
Os2—Os5	2.828 (1)	Os3—C11	2.407 (6)
Os2—Os1—Os4	59.08 (4)	Os1—Os4—Os2	60.10 (4)
Os1—Os2—Os3	75.92 (4)	Os1—Os4—Os3	75.38 (4)
Os1—Os2—Os4	60.82 (4)	Os2—Os4—Os3	61.00 (4)
Os3—Os2—Os4	61.52 (4)	Os1—Os4—Os5	116.97 (5)
Os1—Os2—Os5	117.80 (5)	Os2—Os4—Os5	59.98 (4)
Os3—Os2—Os5	60.35 (4)	Os3—Os4—Os5	60.12 (4)
Os4—Os2—Os5	60.15 (4)	Os2—Os5—Os3	61.58 (4)
Os2—Os3—Os4	57.47 (3)	Os2—Os5—Os4	59.88 (4)
Os2—Os3—Os5	58.07 (3)	Os3—Os5—Os4	61.87 (4)
Os4—Os3—Os5	58.02 (3)	Os1—C11—Os3	93.5 (2)

The hydride ligands were placed in positions calculated with the HYDEX program (Orpen, 1980), but were not refined.

For both compounds, data collection: CAD-4 Software (Enraf–Nonius, 1989); cell refinement: CAD-4 Software; data reduction: NRCVAX DATRD2 (Gabe, Le Page, Charland, Lee & White, 1989); program(s) used to solve structures: NRCVAX SOLVER; program(s) used to refine structures: CRYSTALS (Watkin, Carruthers & Betteridge, 1984); molecular graphics: ORTEPII (Johnson, 1976); software used to prepare material for publication: NRCVAX TABLES.

The authors would like to thank the editor for pointing out the approximate molecular non-crystallographic twofold axis.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1152). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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