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SHORT STRUCTURAL PAPERS

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Dodecacarbonyltetra-μ-hydrido-*tetrahedro*-tetraosmium

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Abstract. $[Os_4(CO)_{12}H_4]$, triclinic, $P\bar{1}$, $a = 9\cdot811$ (3), $b = 9\cdot893$ (3), $c = 10\cdot240$ (4) Å, $\alpha = 85\cdot56$ (2), $\beta = 82\cdot71$ (2), $\gamma = 88\cdot71$ (2)°, $U = 982\cdot82$ Å^3, $Z = 2$, $D_c = 3\cdot72$ Mg m^{-3}, $\mu(Mo\ K\alpha) = 25\cdot83$ mm^{-1}. The structure refined to $R = 0\cdot043$ for 1815 unique diffractometer data. The Os atoms define a distorted tetrahedron with approximate D_{2d} symmetry, there being four long and two short metal–metal bonds. Each Os atom is coordinated to three terminal carbonyls. The arrangement of these groups indicates that the four hydrides edge-bridge the four long Os–Os bonds. The molecules pack in a disordered manner about a molecular centre of symmetry in a ratio of 19 : 1.

Introduction. A knowledge of the molecular geometry of the hydrido-carbonyl clusters of the type

$M_4(CO)_{12}H_4$ (M = Re, Ru, Os) is of importance because these compounds are the starting materials for many of the reactions which are currently being investigated to establish the role of metal clusters in catalysis (Muetterties, 1977; Muetterties, Rhodin, Band, Brucker & Pretzer, 1979). These structures are also of interest from a theoretical viewpoint since $M_4(CO)_{12}H_4$ compounds (M = Ru, Os) have the correct number of electrons to form six two-centre two-electron bonds, but $Re_4(CO)_{12}H_4$ is formally unsaturated, and the location of the hydrides is different in the two types of complex (Hoffmann, Schilling, Bau, Kaesz & Mingos, 1978). IR spectral studies have shown that $Ru_4(CO)_{12}H_4$ and $Os_4(CO)_{12}H_4$ have the same symmetry (Knox, Koepke, Andrews & Kaesz, 1975) while the symmetry of $Re_4(CO)_{12}H_4$ is higher. The structural differences between $Re_4(CO)_{12}H_4$ and $Ru_4(CO)_{12}H_4$ have been confirmed by single-crystal X-ray analyses. In $Re_4(CO)_{12}H_4$ (Wilson & Bau, 1976) the metal atoms define a regular tetrahedron and the 12 carbonyl ligands are eclipsed with respect to the Re—Re edges; this suggests that the four hydrides cap the four faces of the tetrahedron. In $Ru_4(CO)_{12}H_4$ (Wilson, Wu, Love & Bau, 1978) the Ru_4 tetrahedron is distorted to D_{2d} symmetry, with four long and two short Ru—Ru bonds, and the carbonyl groups are staggered with respect to the metal—metal bonds which suggests that the hydrides may bridge the four long tetrahedral edges. There have been many attempts to grow suitable single crystals of $Os_4(CO)_{12}H_4$ so that the similarity between the Os and Ru analogues could be confirmed, and the bond parameters for the parent cluster hydride established. However, because of disorder problems suitable crystals have proved elusive until recently. Single crystals have now been grown and this X-ray analysis was undertaken.

Crystals of $Os_4(CO)_{12}H_4$ were obtained as pale yellow, elongated rectangular blocks by slow evaporation (1 month) from chloroform. 1945 intensities were measured (for $3 \cdot 0 < 2\theta \leq 50 \cdot 0°$) on a Philips PW 1100 four-circle diffractometer, using graphite-monochromated Mo $K\alpha$ radiation, an ω–2θ scan technique, and a crystal $0 \cdot 096 \times 0 \cdot 088 \times 0 \cdot 128$ mm. Lp corrections and semi-empirical absorption corrections (based on a pseudo-ellipsoid model and 440 azimuthal scan data from 20 independent reflections which gave transmission coefficients ranging from $0 \cdot 378$ to $0 \cdot 995$ for the full data set) were applied. Equivalent reflections were averaged to give 1815 unique observed reflections $[F > 3\sigma(F)]$. Cell constants were derived from angular measurements of 25 strong reflections in the range $15 < 2\theta < 25°$.

An E map, calculated using multisolution Σ_2 sign-expansion techniques, showed four strong peaks which defined a tetrahedron; these were considered to be the Os atoms. A subsequent difference synthesis revealed the positions of the carbonyl groups, but also showed four peaks of height *ca* $7 \cdot 8$ e Å^{-3} over the four faces of the Os tetrahedron related to the Os atom positions by an approximate centre of symmetry. These peaks had too high an electron density to be hydride ligands, and there were chemically unreasonable contact distances to the carbonyl groups. An examination of the bond parameters suggested that these peaks represented another orientation of the Os_4 tetrahedron occurring in a small fraction of the sites. The structure was refined by full-matrix least squares. The Os atoms in the two orientations were assigned occupancies k and $1 - k$ respectively; k refined to $0 \cdot 954$ (3). The high-occupancy Os atoms were assigned anisotropic thermal parameters, while the low-occupancy Os atoms, and the C and O atoms (occupancies set to unity) were refined isotropically. A weak constraint was placed on the low-occupancy Os—Os distances, so that the four long and two short bonds in the tetrahedron were each equal; the Os—Os distances refined to values of $2 \cdot 90$ (2) and $2 \cdot 84$ (2) Å. Complex neutral-atom scattering factors (*International Tables for X-ray Crystallography*, 1974), and a weighting scheme $w = 1 \cdot 551/[\sigma^2(F) + 0 \cdot 0002|F|^2]$ were employed. The refinement converged to $R = 0 \cdot 043$ and $R^1 = \sum w^{1/2} \Delta / \sum w^{1/2} |F_o| = 0 \cdot 044$. Final atomic coordinates and isotropic or equivalent isotropic temperature factors are given in Table 1, while the corresponding bond lengths and angles are listed in Tables 2 and 3 respectively.*

Discussion. This X-ray analysis confirms that $Os_4(CO)_{12}H_4$ is isostructural with its Ru analogue (Wilson *et al.*, 1978). The four Os atoms define a distorted tetrahedron with four long [mean $2 \cdot 964$ (2) Å] and two short [mean $2 \cdot 817$ (2) Å] metal—metal bonds. The two short bonds are opposite edges of the tetrahedron so that the cluster core has approximate D_{2d} symmetry. The 12 carbonyl groups are terminal and linear with Os—C and C—O distances of $1 \cdot 91$ (2) and $1 \cdot 14$ (2) Å. These groups are staggered with respect to the Os—Os bonds and the carbonyl polyhedron may be described as a cubo-octahedron. An examination of the *cis* Os—Os—C bond angles shows that the CO groups *cis* to the long Os—Os bonds bend away from them [mean 105 (4)°] while those *cis* to the short bonds do not [mean 94 (2)°]. This suggests that the four hydrides bridge the four long Os—Os edges, the steric influence of the hydrides causing the carbonyl ligands to bend away. Similar trends have been observed in the structure of $Os_3W(CO)_{11}$-$H_3(C_5H_5)$ (Churchill & Hollander, 1979). Fig. 1 shows

Table 1. *Atom coordinates* ($\times 10^4$) *and equivalent isotropic or isotropic temperature factors* ($\text{Å}^2 \times 10^3$)

	x	y	z	U
Os(1)	3103 (1)	2570 (1)	785 (1)	35 (7)*
Os(2)	746 (1)	3127 (1)	2542 (1)	32 (6)*
Os(3)	3476 (1)	3396 (1)	3438 (1)	33 (7)*
Os(4)	2510 (1)	770 (1)	3231 (1)	32 (6)*
Os(1')	1780 (20)	2552 (25)	4170 (18)	46 (7)
Os(2')	4249 (18)	1940 (21)	2434 (18)	32 (6)
Os(3')	1643 (23)	1488 (22)	1635 (19)	44 (7)
Os(4')	2509 (19)	4241 (18)	1826 (18)	32 (6)
C(11)	2051 (28)	1769 (29)	−391 (25)	48 (7)
O(11)	1438 (24)	1216 (23)	−1003 (21)	77 (6)
C(12)	2994 (30)	4235 (31)	−96 (27)	55 (7)
O(12)	3047 (25)	5194 (25)	−838 (22)	83 (7)
C(13)	4808 (34)	2069 (34)	−58 (30)	65 (8)
O(13)	5888 (26)	1684 (25)	−492 (22)	87 (7)
C(21)	−537 (27)	2281 (27)	1589 (24)	45 (7)
O(21)	−1300 (22)	1818 (21)	1013 (19)	66 (6)
C(22)	399 (30)	4845 (30)	1689 (27)	52 (7)
O(22)	243 (21)	5904 (22)	1173 (18)	61 (5)
C(23)	−563 (32)	3416 (32)	4043 (29)	60 (8)
O(23)	−1367 (23)	3647 (23)	4921 (21)	77 (6)
C(31)	5284 (33)	2707 (32)	3617 (28)	59 (8)
O(31)	6374 (26)	2354 (26)	3716 (23)	85 (7)
C(32)	2965 (29)	3362 (30)	5261 (27)	53 (7)
O(32)	2557 (23)	3328 (23)	6377 (20)	73 (6)
C(33)	3958 (32)	5258 (31)	3282 (28)	56 (7)
O(33)	4248 (26)	6368 (27)	3207 (23)	86 (7)
C(41)	4229 (32)	−117 (31)	3407 (27)	57 (8)
O(41)	5296 (25)	−623 (25)	3438 (22)	78 (7)
C(42)	1914 (26)	447 (26)	5087 (24)	40 (6)
O(42)	1590 (22)	229 (22)	6162 (19)	68 (6)
C(43)	1663 (31)	−855 (32)	2801 (28)	59 (8)
O(43)	1206 (23)	−1771 (23)	2492 (20)	75 (6)

* $U_{eq} = \frac{1}{3}$ of the trace of the orthogonalized U matrix.
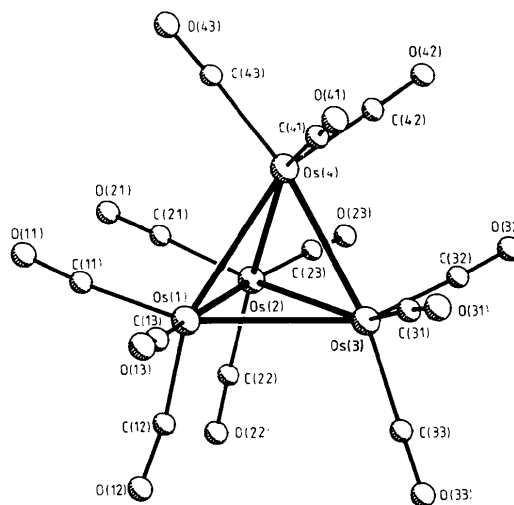
Table 2. *Bond lengths* (Å)

Os(2)−Os(1)	2·816 (2)	C(11)−Os(1)	1·909 (25)
Os(3)−Os(1)	2·965 (2)	C(12)−Os(1)	1·824 (32)
Os(4)−Os(1)	2·962 (2)	C(13)−Os(1)	1·857 (34)
Os(3)−Os(2)	2·963 (2)	C(21)−Os(2)	1·929 (24)
Os(4)−Os(2)	2·967 (2)	C(22)−Os(2)	1·895 (32)
Os(4)−Os(3)	2·817 (2)	C(23)−Os(2)	1·909 (30)
Os(2')−Os(1')	2·899 (22)	C(31)−Os(3)	1·911 (34)
Os(3')−Os(1')	2·895 (21)	C(32)−Os(3)	1·868 (27)
Os(4')−Os(1')	2·839 (24)	C(33)−Os(3)	1·902 (30)
Os(3')−Os(2')	2·837 (24)	C(41)−Os(4)	1·906 (33)
Os(4')−Os(2')	2·891 (22)	C(42)−Os(4)	1·920 (24)
Os(4')−Os(3')	2·900 (22)	C(43)−Os(4)	1·931 (29)
O(11)−C(11)	1·104 (28)	O(31)−C(31)	1·132 (37)
O(12)−C(12)	1·166 (35)	O(32)−C(32)	1·160 (30)
O(13)−C(13)	1·165 (36)	O(33)−C(33)	1·135 (34)
O(21)−C(21)	1·134 (27)	O(41)−C(41)	1·153 (36)
O(22)−C(22)	1·152 (34)	O(42)−C(42)	1·113 (28)
O(23)−C(23)	1·152 (32)	O(43)−C(43)	1·105 (29)

Table 3. *Bond angles* (°)

Os(3)−Os(1)−Os(2)	61·6 (1)	C(11)−Os(1)−Os(2)	92·5 (8)
Os(4)−Os(1)−Os(2)	61·7 (1)	C(11)−Os(1)−Os(3)	151·6 (8)
Os(4)−Os(1)−Os(3)	56·8 (1)	C(11)−Os(1)−Os(4)	102·1 (8)
Os(3)−Os(2)−Os(1)	61·7 (1)	C(12)−Os(1)−Os(2)	91·4 (9)
Os(4)−Os(2)−Os(1)	61·5 (1)	C(12)−Os(1)−Os(3)	99·8 (8)
Os(4)−Os(2)−Os(3)	56·7 (1)	C(12)−Os(1)−Os(4)	149·9 (8)
Os(2)−Os(3)−Os(1)	56·7 (1)	C(13)−Os(1)−Os(2)	168·1 (10)
Os(4)−Os(3)−Os(1)	61·6 (1)	C(13)−Os(1)−Os(3)	108·3 (9)
Os(4)−Os(3)−Os(2)	61·7 (1)	C(13)−Os(1)−Os(4)	107·9 (11)
C(12)−Os(1)−C(11)	91·8 (12)	C(21)−Os(2)−Os(1)	95·9 (7)
C(13)−Os(1)−C(11)	95·8 (12)	C(21)−Os(2)−Os(3)	153·3 (8)
C(13)−Os(1)−C(12)	96·9 (13)	C(21)−Os(2)−Os(4)	100·9 (8)
C(22)−Os(2)−C(21)	91·1 (12)	C(22)−Os(2)−Os(1)	94·7 (9)
C(23)−Os(2)−C(21)	94·9 (11)	C(22)−Os(2)−Os(3)	104·4 (8)
C(23)−Os(2)−C(22)	93·3 (13)	C(22)−Os(2)−Os(4)	154·1 (9)
C(23)−Os(2)−Os(3)	105·6 (9)	C(23)−Os(2)−Os(1)	166·3 (9)
C(32)−Os(3)−C(31)	93·3 (12)	C(23)−Os(2)−Os(4)	108·2 (10)
C(33)−Os(3)−C(32)	93·7 (12)	C(33)−Os(3)−C(31)	95·8 (13)
C(31)−Os(3)−Os(1)	101·2 (8)	C(31)−Os(3)−Os(2)	150·5 (8)
C(31)−Os(3)−Os(4)	91·4 (9)	C(32)−Os(3)−Os(1)	151·7 (8)
C(32)−Os(3)−Os(2)	100·1 (9)	C(32)−Os(3)−Os(4)	94·3 (8)
C(33)−Os(3)−Os(1)	108·6 (8)	C(33)−Os(3)−Os(2)	109·2 (9)
C(33)−Os(3)−Os(4)	168·9 (9)	C(41)−Os(4)−Os(1)	103·4 (9)
C(42)−Os(4)−C(41)	91·6 (11)	C(41)−Os(4)−Os(2)	153·9 (9)
C(43)−Os(4)−C(41)	93·4 (13)	C(42)−Os(4)−Os(1)	152·5 (8)
C(43)−Os(4)−C(42)	93·1 (11)	C(42)−Os(4)−Os(2)	101·1 (8)
O(11)−C(11)−Os(1)	174·4 (27)	C(42) Os(4) Os(3)	94·6 (7)
O(12)−C(12)−Os(1)	168·4 (26)	C(43)−Os(4)−Os(1)	108·5 (9)
O(13)−C(13)−Os(1)	174·2 (32)	C(43)−Os(4)−Os(2)	108·3 (10)
O(21)−C(21)−Os(2)	178·1 (25)	C(43)−Os(4)−Os(3)	168·3 (10)
O(22)−C(22)−Os(2)	177·2 (27)	O(31)−C(31)−Os(3)	177·1 (29)
O(23)−C(23)−Os(2)	176·9 (30)	O(32)−C(32)−Os(3)	175·3 (26)
O(41)−C(41)−Os(4)	175·9 (27)	O(33)−C(33)−Os(3)	179·1 (29)
O(42)−C(42)−Os(4)	178·1 (26)	O(43)−C(43)−Os(4)	176·5 (27)

Fig. 1. The molecular structure of $Os_4(CO)_{12}H_4$.

the molecular geometry and atom-numbering scheme; the disordered low-occupancy Os atoms (denoted Os' in Table 1) have been omitted for clarity.

The D_{2d} symmetry of the tetrahedral core is retained in many of the derivatives of $M_4(CO)_{12}H_4$ (M = Ru, Os). In $|Os_4(CO)_{12}H_2|^{2-}$ (Johnson, Lewis, Raithby,

Sheldrick & Süss, 1978) there are four short and two long metal−metal bonds; the unbridged edges are similar in length [mean of four 2·798 (9) Å] to those in $Os_4(CO)_{12}H_4$, but the two bridged edges are significantly shorter [mean of two 2·934 (4) Å]. This difference is probably due to delocalization of the additional negative charge over the cluster framework, since there is better agreement between the Os−Os bond lengths in $Os_4(CO)_{12}H_4$ and the neutral derivative $Os_4(CO)_{11}H_4(CNCH_3)$ [mean bridged Os−Os

2·963 (2) Å, unbridged 2·822 (1) Å] (Churchill & Hollander, 1980).

The presence of edge-bridging hydrides in $Os_4(CO)_{12}H_4$ is as expected from simple electron-counting considerations. This complex is an 'electron precise' 60 electron system with the correct number of electrons to form two two-centre two-electron Os–Os bonds and four three-centre two-electron Os–H–Os bonds. $Re_4(CO)_{12}H_4$ has only 56 electrons, and it appears to be more favourable to form four four-centre two-electron Re_3H bonds; hence the difference in structure between $Re_4(CO)_{12}H_4$ and $Os_4(CO)_{12}H_4$.

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Di-μ₃-selenido-tris(tricarbonylosmium)(2Os–Os)

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Abstract. $[Os_3(CO)_9Se_2]$, triclinic, $P\bar{1}$, $a = 6\cdot804$ (3), $b = 9\cdot620$ (5), $c = 13\cdot527$ (6) Å, $\alpha = 94\cdot206$ (21), $\beta = 95\cdot570$ (24), $\gamma = 110\cdot469$ (25)°, $U = 820\cdot1$ Å³, $Z = 2$, $D_c = 3\cdot968$ Mg m⁻³, μ(Mo $K\alpha$) = 27·62 mm⁻¹. The structure refined to $R = 0\cdot042$ for 4106 unique observed diffractometer data. The Os atoms define a triangle with one long non-bonding edge [Os···Os 3·791 (1) Å]. The Se atoms cap this triangle on both sides to give a trigonal bipyramidal cluster core. Three terminal carbonyl groups are also bonded to each metal.

Introduction. In trinuclear and square-planar tetranuclear clusters which are capped by ligands on both sides there is the possibility of a bonding interaction between the ligands. In the structure of $Co_4(\mu_2\text{-}CO)_2(CO)_8(\mu_4\text{-}PPh)_2$ (Ryan & Dahl, 1975) the P···P distance of 2·544 (3) Å indicates the presence of a bonding interaction between these atoms. There is less evidence for such an interaction in the S analogue $Co_4(\mu_2\text{-}CO)_2(CO)_8(\mu_4\text{-}S)_2$ (Wei & Dahl, 1975) or in the complex $Fe_3(CO)_9S_2$ (Wei & Dahl, 1965). During the reaction of $Os_3(CO)_{12}$ with elemental Se under reflux, in *n*-octane, $Os_3(CO)_9Se_2$ was isolated as one of the products (Johnson, Lewis, Lodge, Raithby, Henrick & McPartlin, 1979). It was decided to undertake this crystal-structure analysis in order to determine the molecular parameters, and to establish whether an increase in the size of the capping atom increases ligand–ligand bonding interaction.

Deep-yellow platelets of the title compound were obtained by slow crystallization from hexane. 4948 reflections were measured for $3\cdot0 < 2\theta < 60\cdot0°$ on a Stoe four-circle diffractometer with graphite-mono-chromated Mo $K\alpha$ radiation, an ω–θ scan technique, and a crystal 0·34 × 0·29 × 0·10 mm. Lp corrections and a semi-empirical absorption correction based on a pseudo-ellipsoid model with 292 azimuthal scan data from 12 independent reflections were applied; transmission factors ranged from 0·026 to 0·081. The data