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STRUCTURAL STUDIES ON OSMIUM CARBONYL HYDRIDES

XXVII *. THE HETERONUCLEAR RHODIUM-TRIOSMIUM CLUSTER $(\mu\text{-H})_3Os_3Rh(CO)_9(\eta^6\text{-}C_6H_5Me)$

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Summary

The complex $(\mu\text{-H})_3Os_3Rh(CO)_9(\eta^6\text{-}C_6H_5Me)$, previously synthesized by Shore and Hsu has been subjected to a single-crystal X-ray structural analysis. The complex crystallizes in the centrosymmetric monoclinic space group $C2/m$ (No. 12) with a 14.178(2), b 14.683(3), c 10.205(1) Å, β 92.89(1)°, V 2121.6(6) Å^3 and ρ(calcd) 3.20 g cm^{-3} for mol. wt. 1020.78 and $Z = 4$. Diffraction data were collected with a Syntex $P2_1$ automated four-circle diffractometer (Mo-K_α: 2θ 4.0–50.0°) and the structure was solved and refined to R_F 3.9% for all 1964 independent reflections (R_F 3.2% for those 1726 data with $|F_0| > 3\sigma(|F_0|)$). All non-hydrogen atoms were located as were the three hydride ligands which bridge the three Os–Os bonds. The η^6-toluene ligand is bonded to the rhodium atom and is subject to a three-fold disorder; the entire molecule lies about a site of crystallographic $C_s(m)$ symmetry. The metal–metal distances in the tetrahedral cluster are Os(1)–Os(1′) 2.888(1) Å, Os(1)–Os(2) = Os(1′)–Os(2) = 2.888(1) Å, Os(1)–Rh = Os(1′)–Rh = 2.702(1) Å and Os(2)–Rh 2.703(1) Å.

Introduction

Shore and coworkers have shown that the species $(\mu\text{-H})_2Os_3(CO)_{10}$ provides an excellent starting material for hydrido species containing an Os_3M core [4]. More recently they have found that hydride-rich products can be obtained by the simple expedient of carrying out the reaction under an atmosphere of H_2 [5].

We have previously reported structural studies on a variety of heteronuclear hydridotriosmium clusters containing 1st row transition elements obtained in this

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manner, including $(\mu\text{-H})_2Os_3Co(CO)_{10}(\eta^5\text{-}C_5H_5)$ [6], $(\mu\text{-H})_2Os_3Fe(CO)_{13}$ [7], $(\mu\text{-H})_3Os_3Ni(CO)_9(\eta^5\text{-}C_5H_5)$ [1] and $(\mu\text{-H})_3Os_3Co(CO)_9(\eta^5\text{-}C_5H_5)$ [1]. This last species is of particular interest because it is a 59-electron paramagnetic cluster, one electron short of the typical 60-electron tetrahedral cluster.

Attempts to obtain the analogous Os_3Rh complex (as in eq. 1) gave only very small amounts of an uncharacterized paramagnetic material [3].

$$2(\mu\text{-H})_2Os_3(CO)_{10} + 2(\eta^5\text{-}C_5H_5)Rh(CO)_2 + H_2 \rightarrow$$
$$2(\mu\text{-H})_3Os_3Rh(CO)_9(\eta^5\text{-}C_5H_5) + 6CO \qquad (1)$$

However, where this same reaction was carried out in the presence of a solvent that can act as an η^6-ligand (e.g. toluene), a diamagnetic material was obtained: this is shown herein to be the 60-electron species $(\mu\text{-H})_3Os_3Rh(CO)_9(\eta^6\text{-}C_6H_5CH_3)$. Thus, an $\eta^5\text{-}C_5H_5$ ligand on rhodium has been replaced by an $\eta^6\text{-}C_6H_5CH_3$ ligand. A brief communication on this work has appeared previously [3].

Experimental

A rather opaque dark-red crystal of approximate size 0.12 × 0.2 × 0.3 mm was mounted on our Syntex $P2_1$ diffractometer and data were collected as described previously [8], Table 1. All data were converted to $|F_0|$ values following correction for absorption and for Lorentz and polarization factors. Any reflection with $I(\text{net}) < 0$ was assigned a value of $|F_0| = 0$. No data were rejected.

All calculations were performed using the SUNY-Buffalo modified Syntex XTL system on a Data General NOVA 1200 computer.

The systematic absences $h + k = 2n + 1$ (only) in the monoclinic system are compatible with the following three space groups: $C2$ (No. 5), Cm (No. 8) or $C2/m$ (No. 12). The intensity statistics were rather equivocal, so no distinction between the centrosymmetric space group $C2/m$ and the non-centrosymmetric space groups ($C2$ or Cm) was possible on this basis. The structure was initially solved using MULTAN [9] and difference-Fourier syntheses in the noncentrosymmetric space group Cm. It became clear that the two distinct half-molecules defining the structure in space group Cm were related by symmetry and that the true space group was the centrosymmetric $C2/m$.

Subsequent difference-Fourier maps and full-matrix least-squares refinement indicated that the η^6-toluene ligand exhibited a three-fold disorder. This was dealt with by assigning 1/3 of the normal occupancy to the three partial carbon atoms defining the methyl substituent.

The structure refined smoothly to convergence with the following discrepancy indices.

$$R_F = \left[\frac{\Sigma||F_0| - |F_c||}{\Sigma|F_0|}\right] \times 100 = 3.9\%$$

$$R_{wF} = \left[\frac{\Sigma w(|F_0| - |F_c|)^2}{\Sigma w|F_0|^2}\right]^{1/2} \times 100 = 4.0\%$$

$$\text{GOF} = \left[\frac{\Sigma w(|F_0| - |F_c|)^2}{(\text{NO} - \text{NV})}\right]^{1/2} = 1.048$$

TABLE 1

EXPERIMENTAL DATA FOR THE X-RAY DIFFRACTION STUDY OF $(\mu\text{-H})_3Os_3Rh(CO)_9(\eta^6\text{-}CH_3C_6H_5)$

(A) Crystal parameters at 21°C [a]

crystal system: monoclinic
space group: $C2/m$ (No. 12)
a 14.178(2) Å
b 14.683(3) Å
c 10.205(1) Å
β 92.89(1)°

V 2121.6(6) $Å^3$
$Z = 4$
mol.wt. 1020.8
ρ(calcd) 3.20 g cm^{-3}
$\mu(\text{Mo-}K_\alpha)$ 197.6 cm^{-1} [b]

(B) Measurement of data

diffractometer: Syntex $P2_1$
radiation: Mo-K_α ($\bar{\lambda}$ 0.71073 Å)
monochromator: highly oriented graphite, equatorial mode ($2\theta_{mono}$ 12.2°)
reflections measured: $\pm h, k, \pm l$ for 2θ 4.0–50.0°
scan type: θ(crystal)–2θ(counter)
scan width: $[2\theta(K_{\alpha_1})-1.0]-[2\theta(K_{\alpha_2})+1.0]°$
scan speed: 2.5°/min
background measurement: stationary crystal and counter at beginning and end of 2θ scan, each for one fourth of the scan time
reflections collected: 4046 total, merged to 1964 symmetry independent reflections
standard reflections: three measured after each 97 reflections (10,0,0; 0,10,0; 006); no decay was observed over the period of data collection

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[a] Unit cell parameters were derived by a least square fit to the setting angles of the unresolved Mo-$K_{\bar{\alpha}}$ components of 24 reflections with 2θ between 20 and 30°. [b] An empirical absorption correction was applied to all data. This was based upon a series of ψ-scans of close-to-axial reflections that had been collected at 10° intervals of ϕ from 0° through 350°. (The crystal was mounted with the ϕ-axis about 8.8° from b^*.) Reflections used for the ψ-scans, their 2θ values and their max/min intensity ratios were as follows: $04\bar{1}$ ($2\theta = 11.79°$; max/min = 2.41), $06\bar{1}$ (17.14°; 2.26), $08\bar{2}$ (23.70°; 2.23), 0,10,0 (27.95°; 2.12) and 0,12,0 (33.70; 2.09).

TABLE 2

POSITIONAL PARAMETERS FOR $(\mu\text{-H})_3Os_3Rh(CO)_9(\eta^6\text{-}CH_3C_6H_5)$

Atom	x	y	z	B_{iso} ($Å^2$)
Os(1)	0.19530(2)	−0.09833(2)	−0.40304(3)	
Os(2)	0.09695(4)	0	−0.20631(5)	
Rh	0.28734(8)	0	−0.21280(11)	
O(11)	0.3827(6)	−0.1114(6)	−0.5357(12)	
O(12)	0.2300(7)	−0.2638(5)	−0.2286(9)	
O(13)	0.0803(6)	−0.1966(6)	−0.6200(8)	
O(21)	−0.1175(7)	0	−0.2231(11)	
O(22)	0.1184(7)	−0.1525(6)	−0.0045(9)	
C(11)	0.3127(8)	−0.1077(7)	−0.4853(12)	
C(12)	0.2190(8)	−0.2033(7)	−0.2941(10)	
C(13)	0.1216(7)	−0.1603(6)	−0.5372(9)	
C(21)	−0.0391(9)	0	−0.2181(12)	
C(22)	0.1116(7)	−0.0933(7)	−0.0798(10)	
C(1)	0.3387(19)	0	0.000(3)	8.9(6)
C(2)	0.3614(12)	0.0891(11)	−0.0522(18)	8.2(4)
C(3)	0.4178(14)	0.0843(11)	−0.1624(20)	8.7(4)
C(4)	0.4479(19)	0	−0.221(3)	9.3(6)
C(1M)	0.304(6)	0	0.122(10)	10.0(21)
C(3M)	0.461(3)	0.153(3)	−0.188(4)	6.1(8)
H(11)	0.174(8)	0	−0.496(12)	2.8(23)
H(12)	0.094(6)	−0.090(5)	−0.313(9)	3.1(18)

TABLE 3

ANISOTROPIC THERMAL PARAMETERS [a] FOR $(\mu\text{-H})_3Os_3Rh(CO)_9(\eta^6\text{-}CH_3C_6H_5)$

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Os(1)	3.068(19)	3.431(17)	3.373(17)	0.325(11)	0.090(11)	−0.081(11)
Os(2)	3.229(24)	3.877(23)	2.767(21)	0	0.185(15)	0
Rh	3.17(5)	5.37(5)	4.04(5)	0	−1.01(4)	0
O(11)	4.1(4)	7.4(5)	9.6(7)	0.9(3)	2.4(4)	−0.3(4)
O(12)	8.4(6)	5.4(4)	6.7(5)	2.0(4)	0.0(4)	2.3(4)
O(13)	5.8(4)	6.4(4)	5.9(4)	0.5(3)	−1.8(3)	−1.7(4)
O(21)	2.9(5)	10.0(8)	4.9(5)	0	−0.3(4)	0
O(22)	8.1(6)	7.0(5)	5.7(4)	1.4(4)	1.4(4)	2.4(4)
C(11)	4.1(5)	4.9(5)	5.0(5)	0.5(4)	0.8(4)	−0.6(4)
C(12)	4.9(5)	5.0(5)	4.0(5)	1.3(4)	−0.0(4)	−0.1(4)
C(13)	4.1(5)	3.5(4)	3.7(4)	1.2(3)	0.8(3)	−0.0(3)
C(21)	2.7(6)	6.0(7)	2.6(5)	0	0.5(4)	0
C(22)	4.1(5)	5.5(5)	3.7(4)	0.5(4)	0.3(4)	−0.6(4)

[a] These anisotropic thermal parameters enter the equation for the calculated structure factor in the form: $\exp\left(-\frac{1}{4}\left(h^2a^{*2}B_{11}+k^2b^{*2}B_{22}+l^2c^{*2}B_{33}+2hka^*b^*B_{12}+2hla^*c^*B_{13}+2klb^*c^*B_{23}\right)\right)$.

Here, NO is the number of observations and NV is the number of variables. These results are based on 134 parameters refined against all 1964 independent reflections (none rejected).

The discrepancy indices for those 1726 reflections with $|F_0| > 3\sigma(|F_0|)$ were R_F 3.2%, R_{wF} 3.8% and GOF = 1.067.

During the calculations the analytical forms for neutral atoms [10a] were corrected for both the $\Delta f'$ and $i\Delta f''$ terms of anomalous dispersion [10b]. The function minimized was $\Sigma w(|F_0| - |F_c|)^2$ with $w = [\{\sigma(|F_0|)\}^2 + (0.02|F_0|^2)]^{-1}$.

Final positional parameters are collected in Table 2; anisotropic thermal parameters are in Table 3.

Discussion

Crystals of $(\mu\text{-H})_3Os_3Rh(CO)_9(\eta^6\text{-}C_6H_5CH_3)$ contain disordered molecular units which are separated by normal Van der Waals distances. Figure 1 shows the scheme used for labeling atoms. Interatomic distances and their estimated standard deviation (e.s.d.) are collected in Table 4 while important interatomic angles are listed in Table 5.

Each disordered molecule in the crystal has crystallographically-imposed C_s symmetry. The crystallographic "asymmetric unit" consists of one "half-molecule" of $(\mu\text{-H})_3OsRh(CO)_9(\eta^6\text{-}C_6H_5CH_3)$; there are only four molecules per unit cell in the space group *C*2/*m*, which has eight equipoints. A three-fold disorder of the methyl group on the toluene ligand, coupled with the intrinsic three-fold symmetry of the $(\mu\text{-H})_3Os_3(CO)_9$ moiety leads to the entire observed ensemble having approximate C_{3v} symmetry. Figure 1 shows the labeled molecular view as observed down the non-crystallographic three-fold axis.

The C_{3v} symmetry in the basal $(\mu\text{-H})_3Os_3(CO)_9$ unit is clearly indicated by the Os–Os bond distances (Os(1)–Os(1′) 2.888(1) Å, Os(1)–Os(2) 2.888(1) Å and

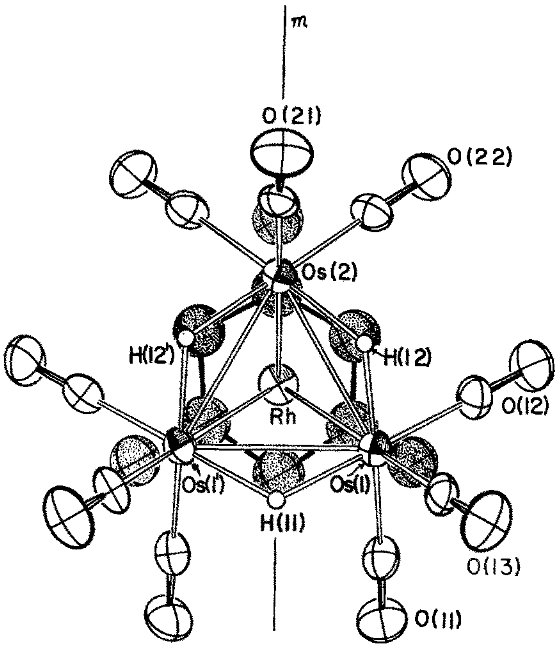


Fig. 1. Labeling diagram and view of the $(\mu\text{-H})_3Os_3Rh(CO)_9(\eta^6\text{-}C_6H_5CH_3)$ molecule down its pseudo three-fold axis. The crystallographic mirror plane is vertical. (ORTEP-II diagram.)

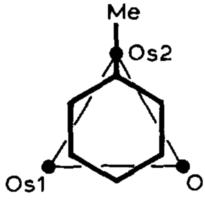
Os(1′)–Os(2) 2.888(1) Å), the Os–Os–Os angles (Os(1′)–Os(1)–Os(2) 60.00(1)°, Os(1)–Os(1′)–Os(2) 60.00(1)° and Os(1)–Os(2)–Os(1′) 59.99(1)°), the disposition of the carbonyl ligands (see Fig. 1) and the uniformity of the bridging hydride ligands

TABLE 4

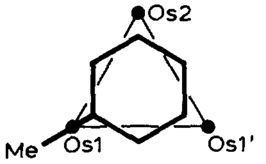
SELECTED INTERATOMIC DISTANCES (Å) FOR THE $(\mu\text{-H})_3Os_3Rh(CO)_9(\eta^6\text{-}CH_3C_6H_5)$ MOLECULE

(A) Metal – metal distances			
Os(1)–Os(1′)	2.888(1)	Os(1)–Os(2)	2.888(1)
Os(1)–Rh	2.702(1)	Os(2)–Rh	2.703(1)
(B) Osmium – carbonyl distances			
Os(1)–C(11)	1.906(11)	Os(1) · · · O(11)	3.048(9)
Os(1)–C(12)	1.920(11)	Os(1) · · · O(12)	3.038(9)
Os(1)–C(13)	1.910(9)	Os(1) · · · O(13)	3.046(8)
Os(2)–C(21)	1.927(13)	Os(2) · · · O(21)	3.036(10)
Os(2)–C(22)	1.887(11)	Os(2) · · · O(22)	3.047(9)
(C) Distances involving the disordered toluene ligand			
Rh–C(1)	2.257(29)	C(1M)–C(1)	1.36(11)
Rh–C(2)	2.308(18)	C(1)–C(2)	1.456(21)
Rh–C(3)	2.264(19)	C(2)–C(3)	1.414(27)
Rh–C(4)	2.282(27)	C(3)–C(3M)	1.22(4)
Rh–Centroid	1.777	C(3)–C(4)	1.450(23)
(D) Metal – hydride distances			
Os(1)–H(11)	1.745(66)	Os(1)–H(12)	1.743(86)
		Os(2)–H(12)	1.719(79)

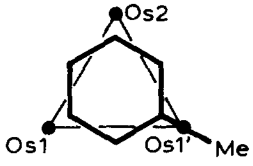
associated with each osmium atom (Os(1)–H(11) = Os(1′)–H(11) = 1.75(7) Å, Os(1)–H(11)–Os(1′) 112(5)°; Os(1)–H(12) 1.74(9) Å, Os(2)–H(12) 1.72(8) Å and Os(1)–H(12)–Os(2) = Os(1′)–H(12)–Os(2) = 113(5)°). The nature of the disorder of the toluene ligand is indicated in structures I–III.



(I)



(II)



(III)

As a result of the basal C_{3v} symmetry, the methyl group has no preference for any one of the above sites, in fact, all three are present in the crystal in equal amount (with each methyl group having a 33% population). The overall observed structure thus has C_{3v} symmetry.

The Rh–Os bond distances are equivalent and consistent with the observed symmetry, with Os(1)–Rh 2.702(1) Å, Os(1′)–Rh 2.702(1) Å and Os(2)–Rh 2.703(1) Å, mean 2.702(1) Å. The Rh–C(arene) bond distances range from 2.257(29) through 2.308(18) Å. The Rh–centroid distance is 1.777 Å. Distances around the arene ring (cyclically) are C(1)–C(2) 1.456(21) Å, C(2)–C(3) 1.414(27) Å, C(3)–C(4) 1.450(23) Å, C(4)–C(3′) 1.450(23) Å, C(3′)–C(2′) 1.414(27) Å and C(2′)–C(1) 1.456(21) Å. The three C(arene)–CH_3 distances are defined by C(1)–C(1M) 1.36(11) Å, C(3)–C(3M) = C(3′)–C(3M′) = 1.22(4) Å. Clearly, there are two possible symmetrical conformations for this disordered ligand relative to the $(\mu\text{-H})_3Os_3(CO)_9$ fragment. The first involves the methyl groups lying directly over the osmium atoms (as is observed with the present structure). The second involves the methyl groups in positions above the centre of each Os–Os bond. We believe that the observed structure (the first conformation) is the favored and more stable arrangement because there is less steric interaction between methyl carbon (and hydrogen) atoms and nearby carbonyl ligands. For example, if the methyl group were to be placed on C(2), a considerably greater steric stress would result with C(22)–O(22) and C(12)–O(12).

The only other osmium–rhodium carbonyl cluster compounds prepared were reported by Stone and coworkers [11]. A crystal structure of $Os_3Rh(\mu\text{-H})_2(acac)(CO)_{10}$ [11] was reported and is the only previous crystal structure of such a mixed-metal cluster compound. The mean value of 2.717(2) Å for the Os–Rh bond distances in $Os_3Rh(\mu\text{-H})_2(acac)(CO)_{10}$ [11] is only 0.015 Å longer than the mean value of 2.702(1) Å observed in the present triosmium-rhodium structure. The osmium–osmium distances in the present structure (vide supra) are similar to hydride-bridged Os–Os distances found in other tetranuclear clusters: viz. 2.893(1)–2.909(1) Å in $(\mu\text{-H})_3Os_3Co(CO)_{12}$ [12], 2.870(1)–2.940(1) Å in $(\mu\text{-H})_2Os_3Co(CO)_{10}(\eta^5\text{-}C_5H_5)$ [6], 2.859(1)–2.874(1) Å in $(\mu\text{-H})_3Os_3Ni(CO)_9(\eta^5\text{-}C_5H_5)$ [1] and 2.831(1)–2.968(1) Å in $Os_3Rh(\mu\text{-H})_2(acac)(CO)_{10}$ [11].

It is interesting that $(\mu\text{-H})_3Os_3Rh(CO)_9(\eta^6\text{-}C_6H_5CH_3)$ is a normal 60-electron tetrahedral cluster while a 60-electron "butterfly" structure is found for $Os_3Rh(\mu\text{-H})_2(acac)(CO)_{10}$.

TABLE 5

SELECTED INTERATOMIC ANGLES (degree) FOR $(\mu\text{-H})_3Os_3Rh(CO)_9(\eta^6\text{-}CH_3C_6H_5)$

(A) Intermetallic angles			
Os(1′)-Os(1)-Os(2)	60.00(1)	Os(1)-Os(2)-Os(1′)	59.99(1)
Os(1′)-Os(1)-Rh	57.70(2)	Os(1)-Os(2)-Rh	57.68(2)
Os(2)-Os(1)-Rh	57.72(2)	Os(1)-Rh-Os(1′)	64.60(2)
		Os(1)-Rh-Os(2)	64.59(2)
(B) M-M-CO and M-M-Centroid angles[a]			
Os(1′)-Os(1)-C(11)	94.12(33)	Os(1)-Os(2)-C(21)	118.26(31)
Os(1′)-Os(1)-C(12)	143.39(32)	Os(1)-Os(2)-C(22)	94.12(32)
Os(1′)-Os(1)-C(13)	118.43(28)	Os(1)-Os(2)-C(22′)	142.94(32)
Os(2)-Os(1)-C(11)	143.60(33)	Rh-Os(2)-C(21)	175.02(31)
Os(2)-Os(1)-C(12)	94.32(32)	Rh-Os(2)-C(22)	86.60(32)
Os(2)-Os(1)-C(13)	118.07(28)	Os(1)-Rh-Centroid	141.73
Rh-Os(1)-C(11)	87.38(33)	Os(2)-Rh-Centroid	142.25
Rh-Os(1)-C(12)	87.07(32)		
Rh-Os(1)-C(13)	175.04(28)		
(C) Carbon-metal-carbon angles			
C(11)-Os(1)-C(12)	93.8(5)	C(21)-Os(2)-C(22)	96.8(5)
C(11)-Os(1)-C(13)	96.2(4)	C(22)-Os(2)-C(22′)	93.2(5)
C(12)-Os(1)-C(13)	96.1(4)		
(D) Metal-carbon-oxygen angles			
Os(1)-C(11)-O(11)	178.5(10)	Os(2)-C(21)-O(21)	179.1(10)
Os(1)-C(12)-O(12)	177.6(10)	Os(2)-C(22)-O(22)	177.7(10)
Os(1)-C(13)-O(13)	177.5(9)		
(E) Angles involved with the hydride ligands			
Os(1)-H(11)-Os(1′)	111.7(51)	Os(1)-H(12)-Os(2)	113.1(45)
C(11)-Os(1)-H(11)	87.2(31)	Os(1′)-Os(1)-H(11)	34.2(31)
C(12)-Os(1)-H(11)	177.5(31)	Os(2)-Os(1)-H(11)	83.6(31)
C(13)-Os(1)-H(11)	86.1(31)	Rh-Os(1)-H(11)	90.7(31)
C(11)-Os(1)-H(12)	174.2(27)	Os(1′)-Os(1)-H(12)	86.4(27)
C(12)-Os(1)-H(12)	82.5(27)	Os(2)-Os(1)-H(12)	33.2(27)
C(13)-Os(1)-H(12)	88.6(27)	Rh-Os(1)-H(12)	88.0(27)
C(21)-Os(2)-H(12)	88.4(28)	Os(1)-Os(2)-H(12)	33.7(27)
C(22)-Os(2)-H(12)	82.4(28)	Os(1′)-Os(2)-H(12)	86.8(27)
C(22′)-Os(2)-H(12)	173.5(28)	Rh-Os(2)-H(12)	88.5(27)
H(11)-Os(1)-H(12)	96.4(41)	H(12)-Os(2)-H(12′)	101.7(39)
(F) Angles involved with the toluene ligand			
C(1M)-C(1)-C(2)[b]	115.6(37)	C(2)-C(3)-C(3M)	116.3(25)
C(2′)-C(1)-C(2)	128.0(19)	C(2)-C(3)-C(4)	124.2(18)
C(1)-C(2)-C(3)	113.1(17)	C(3M)-C(3)-C(4)	117.1(25)
		C(3)-C(4)-C(3′)	117.3(19)

[a] Centroid of the benzene fragment of the disordered toluene ligand. [b] (′) atoms are symmetry related atom, related to the basic asymmetric unit by $[x, -y, z]$.

Although the hydride ligands are located with rather poor precision (as expected with Z(H) = 1 versus Z(Os) = 76), one may easily observe their effects on the ligand distribution about the tetrahedral metal cluster. The expanded equatorial Os-Os-CO angles (due to the stereochemical influence of the hydride ligand) are: Os(1′)-Os(1)-C(13) 118.93(28)°, Os(2)-Os(1)-C(13) 118.07(28)°, and Os(1)-Os(2)-C(21) = Os(1′)-Os(2)-C(21) = 118.26(31) Å.

Each osmium atom has a slightly distorted octahedral environment of ligands if we neglect Os-Os interactions. This geometry is illustrated by the linearity of the *trans*-Rh–Os–C and H–Os–C angles and the orthogonality of the *cis*-C–Os–C, C–Os–H, C–Os–Rh, H–Os–Rh and H–Os–H angles (around Os(1): Rh–Os(1)–C(13) 175.04(28)°, H(12)–Os(1)–C(11) 174.2(27)°, H(11)–Os(1)–C(12) 177.5(31)°; C(11)–Os(1)–C(12) 93.8(5)°, C(11)–Os(1)–C(13) 96.4(2)°, C(12)–Os(1)–C(13) 96.1(4)°, C(11)–Os(1)–H(11) 87.2(31)°, C(13)–Os(1)–H(11) 86.1(31)°, C(12)–Os(1)–H(12) 82.5(27), C(13)–Os(1)–H(12) 88.6(27)°, C(11)–Os(1)–Rh 87.38(33)°, C(12)–Os(1)–Rh 87.07(32)°, H(11)–Os(1)–Rh 90.7(31)°, H(12)–Os(1)–Rh 86.0(27)°, H(11)–Os(1)–H(12) 96.4(41)°. Around Os(2): Rh–Os(2)–C(21) 175.02(31)°, H(12)–Os(2)–C(22′) = H(12′)–Os(2)–C(22) = 173.5(28)°, C(22)–Os(2)–C(21) = C(22′)–Os(2)–C(21) = 96.8(5)°, C(22)–Os(2)–C(22′) 93.2(5)°, C(21)–Os(2)–H(12) = C(22′)–Os(2)–H(12′) = 88.4(28)°, C(22)–Os(2)–H(12) = C(22)–Os(2)–H(12′) = 82.4(28)°, C(22)–Os(2)–Rh = C(22′)–Os(2)–Rh = 86.60(32)°, H(12)–Os(2)–Rh = H(12′)–Os(2)–Rh = 88.5(27)°, H(12)–Os(2)–H(12′) 101.7(39)°).

The Os–C–O systems are all close to linear with individual angles lying between 177.5(9) and 179.1(10)°. Os–C distances range from 1.887(11) through 1.927(13) Å and Os · · · O distances from 3.036(10) through 3.048(9) Å.

Additional material

A listing of observed and calculated structure factor amplitudes is available upon request from M.R.C.

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