Data collection

Syntex $P2_1$ diffractometer
$\theta/2\theta$ scans
Absorption correction: none
2105 measured reflections
1962 independent reflections
1444 observed reflections [$I > 2\sigma(I)$]

$R_{\text{int}} = 0.025$
$\theta_{\max} = 27.5°$
$h = 0 \rightarrow 6$
$k = 0 \rightarrow 13$
$l = -21 \rightarrow 21$
2 standard reflections
  frequency: 120 min
  intensity variation: 5%

Refinement

Refinement on F
Final $R = 0.041$
$wR = 0.059$
$S = 1.36$
1444 reflections
294 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F) + 0.0009|F|^2]$

$(\Delta/\sigma)_{\max} = 0.13$
$\Delta\rho_{\max} = 0.35$ e Å^{-3}
$\Delta\rho_{\min} = -0.40$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$B_{\text{eq}} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
Cu(1)	0.1039 (1)	−0.2480 (1)	0.2161 (1)	2.83 (1)
O(1)	−0.1780 (5)	−0.2785 (2)	0.1362 (1)	3.60 (7)
O(2)	0.4143 (4)	−0.2266 (3)	0.2907 (1)	3.81 (7)
O(3)	0.7881 (5)	−0.1196 (3)	0.3038 (2)	4.16 (7)
N(1)	0.2356 (5)	−0.0998 (3)	0.1616 (2)	2.81 (7)
N(2)	0.0420 (6)	−0.4207 (3)	0.2669 (2)	3.31 (7)
N(3)	0.2020 (6)	−0.4724 (4)	0.3278 (2)	3.85 (8)
C(1)	−0.2141 (6)	−0.2190 (3)	0.0655 (2)	2.90 (9)
C(2)	−0.0533 (7)	−0.1147 (4)	0.0397 (2)	3.24 (9)
C(3)	−0.1013 (8)	−0.0618 (5)	−0.0396 (2)	3.98 (11)
C(4)	−0.3071 (8)	−0.1062 (5)	−0.0910 (2)	4.19 (10)
C(5)	−0.4691 (7)	−0.2047 (5)	−0.0648 (2)	4.30 (12)
C(6)	−0.4283 (6)	−0.2600 (5)	0.0123 (2)	3.96 (9)
C(7)	0.1601 (7)	−0.0604 (4)	0.0903 (2)	3.16 (9)
C(8)	0.4583 (7)	−0.0361 (4)	0.2079 (2)	3.12 (9)
C(9)	0.3637 (9)	0.0894 (5)	0.2497 (3)	4.64 (12)
C(10)	0.5647 (6)	−0.1338 (4)	0.2723 (2)	3.18 (9)
C(11)	0.1248 (8)	−0.5913 (5)	0.3479 (3)	4.95 (13)
C(12)	−0.0967 (11)	−0.6207 (6)	0.2980 (3)	6.22 (16)
C(13)	−0.1411 (9)	−0.5094 (5)	0.2485 (3)	4.96 (13)
N(4)	0.8051 (6)	−0.3064 (4)	0.4377 (2)	4.58 (8)
N(5)	0.6095 (7)	−0.3922 (5)	0.4469 (2)	5.82 (13)
C(14)	0.6904 (10)	−0.4582 (7)	0.5134 (3)	6.25 (16)
C(15)	0.9322 (9)	−0.4226 (6)	0.5437 (3)	5.42 (14)
C(16)	1.0072 (10)	−0.3225 (6)	0.4961 (3)	5.16 (12)

Table 2. *Selected geometric parameters* (Å, °)

Cu(1)—O(1)	1.890 (2)	C(7)—N(1)	1.267 (5)
Cu(1)—O(2)	1.932 (2)	C(10)—O(2)	1.261 (5)
Cu(1)—N(1)	1.890 (3)	C(10)—O(3)	1.220 (4)
Cu(1)—N(2)	1.964 (3)		
O(1)—Cu(1)—N(1)	94.5 (1)	N(1)—Cu(1)—O(2)	84.8 (1)
O(1)—Cu(1)—O(2)	174.2 (1)	N(1)—Cu(1)—N(2)	167.0 (1)
O(1)—Cu(1)—N(2)	90.7 (1)	O(2)—Cu(1)—N(2)	88.9 (1)
Cu—O(1)—C(1)—C(2)	−4.2 (5)	N(1)—Cu—O(1)—C(1)	8.2 (3)
O(1)—C(1)—C(2)—C(7)	−2.6 (5)	C(7)—N(1)—C(8)—H(8)	−39.9 (3)
C(1)—C(2)—C(7)—N(1)	2.2 (6)	C(7)—N(1)—C(8)—C(9)	83.3 (4)
C(2)—C(7)—N(1)—Cu	5.0 (5)	N(1)—C(8)—C(10)—O(2)	−19.8 (4)
C(7)—N(1)—Cu—O(1)	−8.6 (3)		

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N(3)—H···N(5)	0.95 (5)	2.14 (6)	2.850 (5)	131 (5)
N(3)—H···O(2)	0.95 (5)	2.47 (7)	2.785 (5)	99 (4)
N(4)—H···O(3)	0.93 (5)	1.96 (5)	2.890 (5)	174 (4)

All calculations were performed with a local version of the NRC program system (*NRC Crystallographic Programs for the IBM360 System*, 1973).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, least-squares-planes data and all bond lengths have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71245 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: DU1037]

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Structure of the Ethylidene Cluster $Os_3(\mu\text{-}H)_2(\mu\text{-}CHCH_3)(CO)_9(P^iPr_3)$

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Abstract

The title complex, nonacarbonyl-$1\kappa^3C,2\kappa^3C,3\kappa^3C$-$\mu$-ethylidene-$2{:}3\kappa^2C$-di-$\mu$-hydrido-$1{:}2\kappa^2H;2{:}3\kappa^2H$-triisopropylphosphine-$1\kappa P$-*triangulo*-triosmium-(3 *Os—Os*), contains an irregular Os_3 triangle [Os—Os = 3.088 (1)–2.797 (1) Å], with an ethylidene and one hydrido ligand bridging two Os atoms, and a P^iPr_3 ligand coordinated in an equatorial position to the third Os atom. A second bridging hydride is

coordinated *cis* to the phosphine ligand. The locations proposed for the two hydrides are based on NMR evidence and potential-energy calculations.

Comment

The related methylene cluster $Os_3(\mu\text{-}H)_2(\mu\text{-}CH_2)(CO)_{10}$ has been characterized by single-crystal neutron diffraction (Schultz, Williams, Calvert, Shapley & Stucky, 1979), and has a very similar structure to the title complex, but with slightly different Os—Os distances [Os—Os = 3.053 (3)–2.824 (3) Å]. The unsubstituted derivative $Os_3(\mu\text{-}H)_2(\mu\text{-}CHCH_3)(CO)_{10}$ has been spectroscopically characterized (Cree-Uchiyama, Shapley & St Georges, 1986) and the ^1H NMR parameters are very similar to those of the title complex. $Os_3(\mu\text{-}H)_2(\mu\text{-}CHCH_3)(CO)_{10}$ exists in tautomeric equilibrium with the agonistic ethyl complex $Os_3(\mu\text{-}H)(\mu\text{-}C_2H_5)(CO)_{10}$ (Cree-Uchiyama *et al.*, 1986). In a similar fashion, $Os_3(\mu\text{-}H)_2(\mu\text{-}CH_2)(CO)_{10}$ exists in tautomatic equilibrium with the agonistic methyl complex $Os_3(\mu\text{-}H)(\mu\text{-}CH_3)(CO)_{10}$ (Calvert & Shapley, 1977, 1978). In contrast, the NMR evidence indicates that the title cluster exists solely as the stable ethylidene tautomer. This may be a result of the relatively greater electron density in the title cluster favouring the better π acid $\mu\text{-}CHCH_3$.

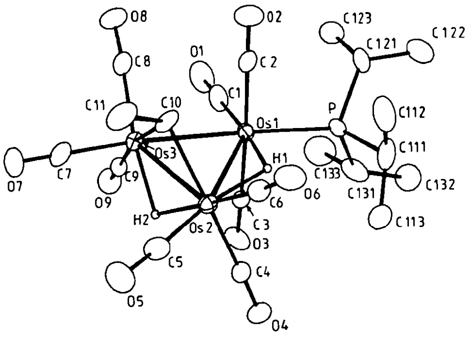

Fig. 1. Molecular structure and atomic labelling scheme for $Os_3(\mu\text{-}H)_2(\mu\text{-}CHCH_3)(CO)_9(P^iPr_3)$. Thermal ellipsoids are shown at the 20% probability level; two H atoms are shown as spheres of arbitary size; other H atoms are omitted for clarity.

Experimental

Crystal data

$[Os_3(H)_2(C_2H_4)(C_9H_{21}P)(CO)_9]$
$M_r = 1013.0$
Monoclinic
$P2_1/n$
$a = 9.165$ (2) Å
$b = 19.473$ (4) Å
$c = 15.482$ (4) Å
$\beta = 97.51$ (2)°

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 12\text{–}14°$
$\mu = 14.00$ mm^{-1}
$T = 298$ K
Prism
0.7 × 0.35 × 0.3 mm

$V = 2739$ (1) Å^3
$Z = 4$
$D_x = 2.46$ Mg m^{-3}

Yellow
Crystal source: hexane

Data collection

Enraf-Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: by *DIFABS* (Walker & Stuart, 1983)
$T_{min} = 0.73$, $T_{max} = 1.32$
5310 measured reflections
4816 independent reflections
2854 observed reflections $[I > 2.5\sigma(I)]$

$R_{int} = 0.023$
$\theta_{max} = 25°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 23$
$l = -16 \rightarrow 16$
2 standard reflections ($\bar{3}09$, $\bar{2}48$)
frequency: 120 min
intensity variation: 10% over 74 h data collection

Refinement

Refinement on F
Final $R = 0.032$
$wR = 0.035$
$S = 1.49$
2854 reflections
298 parameters
H-atom parameters not refined

$w = [\sigma^2(F_o)]^{-1}$
$(\Delta/\sigma)_{max} = 0.01$
$\Delta\rho_{max} = 1.06$ e Å^{-3}
$\Delta\rho_{min} = -1.17$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters* (Å^2)

U_{iso} for H atoms; $U_{eq} = \frac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$ for other atoms.

	x	y	z	U_{iso}/U_{eq}
Os(1)	0.86651 (7)	0.13641 (3)	0.66809 (3)	0.046
Os(2)	1.08570 (6)	0.24018 (3)	0.76385 (3)	0.048
Os(3)	0.96738 (6)	0.25791 (3)	0.58938 (3)	0.045
P	0.8057 (4)	0.0366 (2)	0.7454 (2)	0.052
O(1)	0.6510 (15)	0.1223 (6)	0.5025 (7)	0.113
O(2)	1.0958 (15)	0.0564 (6)	0.5789 (8)	0.118
O(3)	0.6484 (12)	0.2227 (5)	0.7558 (8)	0.086
O(4)	0.9004 (14)	0.2690 (6)	0.9125 (6)	0.088
O(5)	1.2417 (15)	0.3767 (6)	0.7989 (8)	0.104
O(6)	1.3405 (13)	0.1592 (6)	0.8575 (6)	0.093
O(7)	1.0891 (15)	0.3913 (6)	0.5268 (8)	0.105
O(8)	1.0055 (14)	0.1832 (6)	0.4228 (6)	0.099
O(9)	0.6491 (12)	0.3071 (6)	0.5426 (7)	0.085
C(1)	0.7278 (18)	0.1279 (8)	0.5664 (10)	0.078
C(2)	1.0092 (19)	0.0873 (8)	0.6135 (8)	0.071
C(3)	0.7351 (17)	0.1901 (7)	0.7242 (8)	0.056
C(4)	0.9632 (17)	0.2569 (7)	0.8562 (8)	0.058
C(5)	1.1840 (18)	0.3227 (9)	0.7881 (9)	0.074
C(6)	1.2395 (18)	0.1904 (8)	0.8254 (8)	0.065
C(7)	1.0455 (17)	0.3408 (9)	0.5511 (8)	0.069
C(8)	0.9898 (18)	0.2095 (8)	0.4852 (8)	0.065
C(9)	0.7690 (16)	0.2882 (7)	0.5607 (7)	0.051
C(10)	1.1834 (15)	0.2203 (8)	0.6468 (7)	0.061
C(11)	1.3191 (17)	0.2623 (10)	0.6309 (9)	0.088
C(111)	0.934 (3)	0.022 (1)	0.850 (1)	0.121
C(112)	1.088 (2)	0.006 (1)	0.827 (1)	0.105
C(113)	0.911 (2)	0.077 (1)	0.917 (1)	0.103
C(121)	0.829 (2)	−0.043 (1)	0.690 (1)	0.111
C(122)	0.832 (3)	−0.109 (1)	0.740 (1)	0.169
C(123)	0.713 (3)	−0.048 (1)	0.598 (1)	0.127
C(131)	0.628 (3)	0.041 (1)	0.786 (2)	0.141
C(132)	0.585 (3)	−0.012 (1)	0.847 (1)	0.151
C(133)	0.497 (2)	0.051 (1)	0.700 (2)	0.143
H(1)	1.00640	0.15270	0.76270	0.050
H(2)	0.94690	0.29420	0.69760	0.050

Table 2. *Selected bond lengths* (Å) *and angles* (°)

Os(1)—Os(2)	3.088 (1)	Os(1)—Os(3)	2.869 (1)
Os(1)—P	2.386 (4)	Os(1)—C(1)	1.897 (16)
Os(1)—C(2)	1.905 (16)	Os(1)—C(3)	1.890 (15)
Os(1)—H(1)	1.844 (1)	Os(2)—Os(3)	2.797 (1)
Os(2)—C(4)	1.957 (14)	Os(2)—C(5)	1.856 (17)
Os(2)—C(6)	1.867 (16)	Os(2)—C(10)	2.159 (12)
Os(2)—H(1)	1.851 (1)	Os(2)—H(2)	1.854 (1)
Os(3)—C(7)	1.892 (16)	Os(3)—C(8)	1.903 (14)
Os(3)—C(9)	1.908 (15)	Os(3)—C(10)	2.187 (14)
Os(3)—H(2)	1.850 (1)	P—C(111)	1.90 (2)
P—C(121)	1.792 (17)	P—C(131)	1.82 (3)
Os(2)—Os(1)—Os(3)	55.9 (1)	Os(2)—Os(1)—P	118.2 (1)
Os(2)—Os(1)—C(1)	141.2 (5)	Os(2)—Os(1)—C(2)	95.8 (5)
Os(2)—Os(1)—C(3)	80.3 (5)	Os(2)—Os(1)—H(1)	33.4 (1)
Os(3)—Os(1)—P	173.7 (1)	Os(3)—Os(1)—C(1)	86.5 (5)
Os(3)—Os(1)—C(2)	87.0 (5)	Os(3)—Os(1)—C(3)	90.0 (5)
Os(3)—Os(1)—H(1)	88.1 (1)	P—Os(1)—C(1)	99.8 (5)
P—Os(1)—C(2)	91.9 (5)	P—Os(1)—C(3)	90.9 (5)
P—Os(1)—H(1)	85.7 (1)	C(1)—Os(1)—C(2)	90.7 (7)
C(1)—Os(1)—C(3)	91.8 (7)	C(1)—Os(1)—H(1)	174.5 (5)
C(2)—Os(1)—C(3)	175.9 (7)	C(2)—Os(1)—H(1)	89.6 (5)
C(3)—Os(1)—H(1)	87.6 (5)	Os(1)—Os(2)—Os(3)	58.1 (1)
Os(1)—Os(2)—C(4)	93.5 (5)	Os(1)—Os(2)—C(5)	158.7 (5)
Os(1)—Os(2)—C(6)	107.8 (5)	Os(1)—Os(2)—C(10)	78.2 (4)
Os(1)—Os(2)—H(1)	33.2 (1)	Os(1)—Os(2)—H(2)	75.5 (1)
Os(3)—Os(2)—C(4)	119.9 (5)	Os(3)—Os(2)—C(5)	101.9 (5)
Os(3)—Os(2)—C(6)	136.8 (4)	Os(3)—Os(2)—C(10)	50.4 (4)
Os(3)—Os(2)—H(1)	90.1 (1)	Os(3)—Os(2)—H(2)	40.9 (1)
C(4)—Os(2)—C(5)	91.0 (7)	C(4)—Os(2)—C(6)	100.4 (6)
C(4)—Os(2)—C(10)	169.6 (6)	C(4)—Os(2)—H(1)	84.2 (4)
C(4)—Os(2)—H(2)	83.8 (4)	C(5)—Os(2)—C(6)	91.8 (7)
C(5)—Os(2)—C(10)	94.7 (6)	C(5)—Os(2)—H(1)	167.9 (5)
C(5)—Os(2)—H(2)	84.3 (5)	C(6)—Os(2)—C(10)	88.1 (6)
C(6)—Os(2)—H(1)	78.2 (5)	C(6)—Os(2)—H(2)	174.4 (5)
C(10)—Os(2)—H(1)	91.9 (5)	C(10)—Os(2)—H(2)	88.1 (4)
H(1)—Os(2)—H(2)	106.1 (1)	Os(1)—Os(3)—Os(2)	66.0 (1)
Os(1)—Os(3)—C(7)	173.0 (4)	Os(1)—Os(3)—C(8)	91.6 (5)
Os(1)—Os(3)—C(9)	90.2 (4)	Os(1)—Os(3)—C(10)	82.9 (4)
Os(1)—Os(3)—H(2)	81.6 (1)	Os(2)—Os(3)—C(7)	107.0 (4)
Os(2)—Os(3)—C(8)	133.3 (5)	Os(2)—Os(3)—C(9)	120.0 (4)
Os(2)—Os(3)—C(10)	49.5 (3)	Os(2)—Os(3)—H(2)	41.0 (1)
C(7)—Os(3)—C(8)	94.0 (6)	C(7)—Os(3)—C(9)	92.9 (7)
C(7)—Os(3)—C(10)	93.0 (7)	C(7)—Os(3)—H(2)	92.6 (4)
C(8)—Os(3)—C(9)	99.3 (6)	C(8)—Os(3)—C(10)	89.2 (6)
C(8)—Os(3)—H(2)	172.7 (5)	C(9)—Os(3)—C(10)	169.2 (5)
C(9)—Os(3)—H(2)	83.5 (4)	C(10)—Os(3)—H(2)	87.3 (4)

The title complex was obtained in very low yield (0.5%) from the reaction between $Os_3(\mu\text{-}H)_2(CO)_9(P^iPr_3)$ and $Pt(PCy_3)(C_2H_4)_2$, which was used to prepare $Os_3Pt(\mu\text{-}H)_2(CO)_9$(*Pt*-$PCy_3$)(*Os*-$P^iPr_3$) (Farrugia & Rae, 1991). ^1H NMR data (CD_2Cl_2, 218 K), δ: 6.64 [*q*, 1H, $C\underline{H}CH_3$, *J*(H-H) = 7.3 Hz], 2.45 [*d*, 3H, $CHC\underline{H}_3$, *J*(H-H) = 7.3 Hz], 2.21 [septet, 3H, $C\underline{H}Me_2$, *J*(H-H) = 7.1 Hz], 1.10 [*dd*, 18H, $CH(C\underline{H}_3)_2$, *J*(H-H) = 7.1, *J*(P-H) = 14.9 Hz], −14.35 [*s*, 1H, Os(μ-H)Os], −20.96 p.p.m. [*d*, 1H, Os(μ-H)Os, *J*(P-H) = 9.7 Hz]. $^{31}P\{^1H\}$ NMR data (CD_2Cl_2, 190 K), δ: 26.1 p.p.m. (*s*). No signals from an ethyl tautomer (Cree-Uchiyama *et al.*, 1986) were detected either immediately, or on standing for 12 h.

Systematic absences $k = 2n+1$ in $0k0$, $h+l = 2n+1$ in $h0l$ were observed for the data crystal. Data were corrected for decomposition and Lp effects.

The structure was solved by direct methods (*MITHRIL*; Gilmore, 1984), and subsequent full-matrix least-squares refinement included anisotropic thermal parameters for all non-H atoms. Aliphatic H atoms were included at calculated positions (C—H = 1.0 Å) for the methyl and isopropyl groups with fixed contributions to the structure factors. The methine H atom on the ethylidene group was not observed in difference maps and was not included. The metal hydride positions were determined using potential-energy minimization (*HYDEX*; Orpen, 1980) with Os—H = 1.85 Å. Calculations were carried out on a MicroVAX 3600 computer using the Glasgow *GX* suite of programs (Mallinson & Muir, 1985).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71126 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1037]

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Electrooxidation of the Redox Tertiary Phosphine 4-(Diphenylphosphino)-4′,5,5′-trimethyltetrathiafulvalene (P_1) and the Structure of $(P_1O^{\bullet +})_2Mo_6Br_{14}^{2-}$

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Abstract

Electrooxidation of the diphenyl(4′,5,5′-trimethyl-1,1′,3,3′-tetrathia-4-fulvalenyl)phosphine (hereafter P_1) in the presence of $(^nBu_4N^+)_2Mo_6Br_{14}^{2-}$ affords the cation radical salt diphenyl[4-(4′,5,5′-trimethyl-1,1′,3,3′-tetrathia-4-fulvalenyl)]phosphoniumyl oxide–hexamolybdenum tetradecabromide (2/1),

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