C(2)	0.797 (1)	0.006 (1)	0.181 (1)	6.6 (3)
C(3)	0.3423 (9)	−0.174 (1)	0.2157 (9)	5.0 (2)
C(4)	0.3748 (7)	−0.237 (1)	0.0862 (9)	4.5 (2)
C(5)	0.6217 (8)	−0.286 (1)	0.0634 (9)	4.4 (2)
C(6)	0.7727 (8)	−0.281 (1)	0.0226 (9)	4.7 (2)
C(7)	0.8737 (9)	−0.358 (1)	0.059 (1)	5.2 (2)
C(8)	1.007 (1)	−0.320 (1)	0.002 (2)	5.8 (2)
C(9)	0.9779 (9)	−0.228 (2)	−0.077 (1)	5.6 (2)

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

Cd—Cl	2.533 (3)	Cd—N(1)	2.458 (9)
Cd—N(2)	2.45 (1)	C(1)—N(1)	1.50 (2)
C(1)—C(1^i)	1.55 (2)	N(1)—C(2)	1.44 (2)
N(2)—C(4)	1.48 (1)	N(2)—C(5)	1.33 (1)
N(3)—C(6)	1.36 (2)	N(3)—C(9)	1.39 (1)
C(3)—C(4)	1.51 (1)	C(5)—C(6)	1.45 (1)
C(6)—C(7)	1.35 (2)	C(7)—C(8)	1.43 (2)
C(8)—C(9)	1.35 (3)		
Cl—Cd—Cl^i	95.0 (2)	Cl—Cd—N(1)	95.1 (3)
Cl—Cd—N(1^i)	167.8 (3)	Cl—Cd—N(2)	101.1 (1)
Cl—Cd—N(2^i)	92.2 (2)	N(1)—Cd—N(1^i)	75.8 (5)
N(1)—Cd—N(2)	92.5 (3)	N(1)—Cd—N(2^i)	71.7 (3)
N(2)—Cd—N(2^i)	160.3 (4)	N(1)—C(1)—N(1^i)	112.1 (8)
C(1)—N(1)—C(2)	109 (1)	C(4)—N(2)—C(5)	114 (1)
C(6)—N(3)—C(9)	108 (1)	N(2)—C(4)—C(3)	108.0 (9)
N(2)—C(5)—C(6)	125 (1)	N(3)—C(6)—C(5)	126 (1)
N(3)—C(6)—C(7)	110.0 (9)	C(5)—C(6)—C(7)	124 (1)
C(6)—C(7)—C(8)	106 (1)	C(7)—C(8)—C(9)	108 (1)
N(3)—C(9)—C(8)	108 (1)		

Symmetry code: (i) $1 - x, -y, z$.

Lp corrections were applied to intensity data. The heavy-atom method (Cd atom) followed by difference Fourier syntheses revealed the positions of all non-H atoms. The positions of the H atoms were calculated geometrically and were not refined but included in F_c calculations [C—H = 0.98 Å; B(H) = 1.3B(C)]. A full-matrix least-squares refinement was used with anisotropic thermal parameters for Cd, Cl and N atoms, isotropic for C and H. Calculations were performed on a VAXstation 3100 computer with the *SDP* programs (Frenz, 1985); molecular graphics were produced using *ORTEP* (Johnson, 1965).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71503 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI1057]

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A Series of Osmium Carbonyl Complexes with Related Terminal, Bridging and Capping Phosphorus Ligands

ALEXANDER J. BLAKE,* E. A. V. EBSWORTH,† ALAN P. MCINTOSH AND MARTIN SCHRÖDER

Department of Chemistry, The University of Edinburgh, West Mains Road, Edinburgh EH9 3JJ, Scotland

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Abstract

The structures of a series of three triosmium carbonyl clusters containing closely related phosphorus ligands have been determined: decarbonyl-1κ^3C,2κ^3C,3κ^4C-(dimethylphosphine-1κP)-μ-hydrido-1:2κ^2H-hydrido-2κH-*triangulo*-triosmium(3 Os—Os), [$Os_3(CO)_{10}H(\mu_2\text{-}H)(PMe_2H)$] (1), contains a terminal $PHMe_2$ ligand; decacarbonyl-1κ^3C,2κ^3C,3κ^4C-(μ-dimethylphosphino-1:2κ^2P)-μ-hydrido-1:2κ^2H-*triangulo*-triosmium(3 Os—Os), [$Os_3(CO)_{10}(\mu_2\text{-}H)(\mu_2\text{-}PMe_2)$] (2), has a PMe_2 group bridging two of the osmium centres; in nonacarbonyl-1κ^3C,2κ^3C,3κ^3C-bis-μ-hydrido-2:3κ^2H;1:3κ^2H-(μ_3-methylphosphinidene-κ^3P)-*triangulo*-triosmium-(3 Os—Os), [$Os_3(CO)_9(\mu_2\text{-}H)_2(\mu_3\text{-}PMe)$] (3), the PMe group caps all three metals. In (1) the terminal Os—P distance is 2.316 (18) Å and in (2) the two Os—P distances of 2.360 (4) and 2.365 (4) Å do not differ significantly. However, the μ_3-PMe group in (3) does not cap symmetrically: at 2.350 (5) Å, one of the Os—P distances is significantly longer than the remaining two, 2.318 (5) and 2.317 (5) Å.

Comment

Many structure determinations have been reported of triosmium carbonyl clusters containing terminal phosphines but there are relatively few reports of such clusters with bridging (μ_2) PR_2 ligands

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† Presently Vice-Chancellor, University of Durham, Old Shire Hall, Durham DH1 3HP, England.

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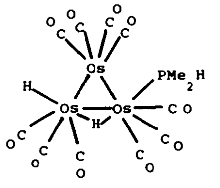
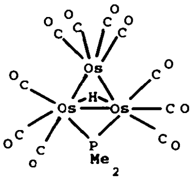
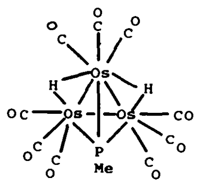
(Huttner, Natarajan & Zsolnai, 1981; Bruce, Guss, Mason, Skelton & White, 1983; Carty, Cherkas, Nucciarone, Patel & Taylor, 1985; Day, Deeming, Doherty, Hardcastle & Minassian, 1991; Carty, Cherkas & Taylor, 1990). There is only one previous report of a capping (μ_3) PR ligand: $[Os_3(CO)_9(\mu_2\text{-}H)_2(\mu_3\text{-}PPh)]$ (Huttner, Natarajan & Zsolnai, 1981),



(1)



(2)



(3)

which shows, as in (3), one long and two short Os—P distances. The structures of analogous ruthenium complexes containing μ_2-PPhH and μ_3-PPh ligands have been determined (Iwasaki, Mays, Raithby, Taylor & Wheatley, 1981). We have described the syntheses of (1), (2) and (3) previously (Ebsworth, McIntosh & Schröder, 1986) and a general route to $[Os_3(CO)_{10}(\mu_2\text{-}H)(\mu_2\text{-}PR^1R^2)]$ has been published (Colbran, Johnson, Lewis & Sorrell, 1985).

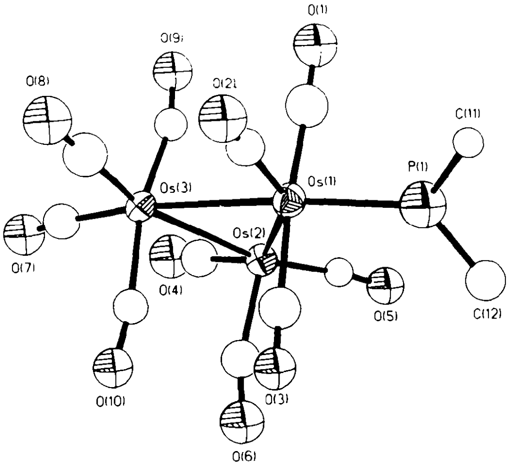


Fig. 1. A view of (1) showing the labelling of the non-H atoms. Thermal ellipsoids are drawn at the 30% probability level.

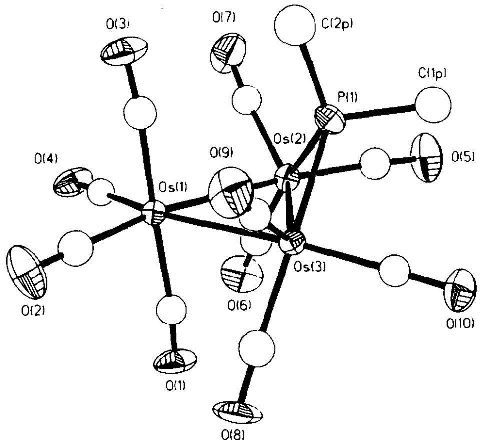


Fig. 2. A view of (2) showing the labelling of the non-H atoms. Thermal ellipsoids are drawn at the 30% probability level.

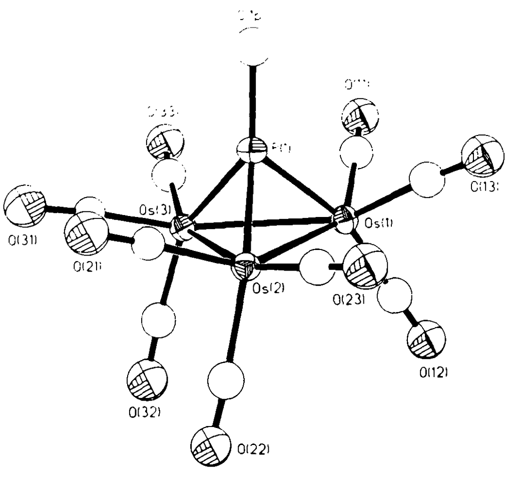


Fig. 3. A view of (3) showing the labelling of the non-H atoms. Thermal ellipsoids are drawn at the 30% probability level.

Experimental

Compound (1) was prepared as described previously (Ebsworth, McIntosh & Schröder, 1986) from the reaction of $[Os_3(CO)_{10}H_2]$ with Me_2PH in PhMe at 298 K, and was recrystallized from PhMe. Compound (2) was prepared by heating $[Os_3(H)_2(C_2H_7P)(CO)_{10}]$ (1) and was recrystallized from PhMe. Compound (3) was prepared by heating $[Os_3H(CH_3P)(CO)_{10}]$ and was recrystallized from PhMe.

Compound (1)

Crystal data

$[Os_3(H)_2(C_2H_7P)(CO)_{10}]$
$M_r = 914.7$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Orthorhombic
$Pn2_1a$
a = 15.995 (5) Å
b = 12.958 (7) Å
c = 9.732 (4) Å
V = 2017 Å^3
Z = 4
D_x = 3.012 Mg m^{-3}

Cell parameters from 14 reflections
θ = 3 - 18°
μ = 19.00 mm^{-1}
T = 298 K
Needle
0.40 × 0.08 × 0.04 mm
Dark red

Data collection

Stoe Stadi-2 two-circle diffractometer
ω scans
Absorption correction: empirical
$T_{\min}$ = 0.93, $T_{\max}$ = 1.11
1997 measured reflections
1997 independent reflections

1056 observed reflections [$F > 6\sigma(F)$]
$\theta_{\max}$ = 25°
h = 0 → 19
k = 0 → 15
l = 0 → 12
No standard reflections

Refinement

Refinement on F
R = 0.0444
wR = 0.0477
S = 1.106
1056 reflections
128 parameters
H atoms not located

$w = 1/[\sigma^2(F) + 0.000429F^2]$
$(\Delta/\sigma)_{\max}$ = 0.02
$\Delta\rho_{\max}$ = 1.13 e Å^{-3}
$\Delta\rho_{\min}$ = −0.97 e Å^{-3}
Atomic scattering factors from Cromer & Mann (1968) or inlaid

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

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$ for Os atoms.

	x	y	z	$U_{\rm iso}/U_{\rm eq}$
Os(1)	−0.10477 (8)	−0.0039 (2)	−0.50385 (16)	0.0589 (8)
Os(2)	−0.27201 (8)	0.103 (2)	−0.50765 (19)	0.0606 (9)
Os(3)	−0.20953 (8)	0†	−0.74705 (19)	0.0569 (7)
P(1)	−0.0424 (8)	0.0082 (19)	−0.2897 (14)	0.109 (4)
C(11)	0.049 (3)	−0.078 (4)	−0.252 (5)	0.042 (8)
C(12)	−0.047 (4)	0.115 (6)	−0.180 (6)	0.084 (9)
C(11′)	−0.093 (6)	0.078 (7)	−0.152 (8)	0.091 (9)
C(12′)	0.011 (5)	−0.097 (6)	−0.225 (7)	0.064 (9)
C(1)	−0.163 (3)	−0.124 (4)	−0.437 (5)	0.100 (8)
O(1)	−0.1938 (19)	−0.206 (3)	−0.400 (4)	0.109 (7)
C(2)	−0.028 (2)	−0.086 (3)	−0.600 (4)	0.068 (7)
O(2)	0.0264 (18)	−0.127 (3)	−0.650 (3)	0.098 (7)
C(3)	−0.055 (3)	0.127 (4)	−0.553 (4)	0.089 (8)
O(3)	−0.0145 (17)	0.194 (3)	−0.582 (3)	0.084 (6)
C(4)	−0.374 (3)	0.110 (4)	−0.603 (4)	0.093 (8)
O(4)	−0.444 (2)	0.116 (4)	−0.646 (4)	0.134 (8)
C(5)	−0.3054 (19)	0.139 (3)	−0.340 (4)	0.055 (7)
O(5)	−0.3311 (17)	0.171 (2)	−0.233 (3)	0.095 (7)
C(6)	−0.238 (3)	0.236 (4)	−0.575 (5)	0.090 (8)
O(6)	−0.210 (2)	0.320 (3)	−0.588 (4)	0.113 (7)
C(7)	−0.291 (3)	0.021 (4)	−0.885 (4)	0.080 (7)
O(7)	−0.345 (2)	0.043 (3)	−0.954 (3)	0.107 (7)
C(8)	−0.133 (3)	−0.085 (4)	−0.852 (5)	0.099 (8)
O(8)	−0.089 (2)	−0.137 (3)	−0.916 (4)	0.110 (7)
C(9)	−0.271 (2)	−0.106 (3)	−0.663 (4)	0.066 (7)
O(9)	−0.3187 (18)	−0.180 (3)	−0.640 (3)	0.097 (7)
C(10)	−0.157 (2)	0.127 (3)	−0.791 (4)	0.066 (7)
O(10)	−0.1267 (18)	0.200 (2)	−0.837 (3)	0.081 (6)

† Coordinate fixed to define origin.

Table 2. *Geometric parameters* (Å, °) *for* (1)

Os(1)—Os(2)	3.012 (3)	P(1)—C(11)	1.88 (5)
Os(1)—Os(3)	2.900 (2)	P(1)—C(12)	1.75 (7)
Os(2)—Os(3)	2.865 (2)	P(1)—C(11′)	1.81 (9)
Os(1)—P(1)	2.316 (18)	P(1)—C(12′)	1.73 (8)
Os(1)—C(1)	1.93 (5)	C(1)—O(1)	1.22 (6)
Os(1)—C(2)	1.88 (4)	C(2)—O(2)	1.13 (5)
Os(1)—C(3)	1.93 (4)	C(3)—O(3)	1.13 (5)
Os(2)—C(4)	1.88 (5)	C(4)—O(4)	1.19 (6)
Os(2)—C(5)	1.77 (3)	C(5)—O(5)	1.20 (5)
Os(2)—C(6)	1.92 (5)	C(6)—O(6)	1.19 (6)
Os(3)—C(7)	1.89 (4)	C(7)—O(7)	1.13 (5)
Os(3)—C(8)	1.94 (5)	C(8)—O(8)	1.15 (6)
Os(3)—C(9)	1.88 (4)	C(9)—O(9)	1.24 (5)
Os(3)—C(10)	1.90 (4)	C(10)—O(10)	1.15 (5)
Os(2)—Os(1)—Os(3)	57.92 (6)	Os(1)—Os(3)—C(9)	86.2 (12)
Os(2)—Os(1)—P(1)	111.2 (5)	Os(1)—Os(3)—C(10)	87.0 (11)
Os(2)—Os(1)—C(1)	87.2 (14)	Os(2)—Os(3)—C(7)	105.7 (13)
Os(2)—Os(1)—C(2)	146.8 (12)	Os(2)—Os(3)—C(8)	156.2 (15)
Os(2)—Os(1)—C(3)	87.6 (13)	Os(2)—Os(3)—C(9)	78.9 (12)
Os(3)—Os(1)—P(1)	169.1 (5)	Os(2)—Os(3)—C(10)	86.2 (11)
Os(3)—Os(1)—C(1)	90.8 (14)	C(7)—Os(3)—C(8)	98.1 (19)
Os(3)—Os(1)—C(2)	89.0 (12)	C(7)—Os(3)—C(9)	93.2 (17)
Os(3)—Os(1)—C(3)	91.2 (13)	C(7)—Os(3)—C(10)	90.9 (17)
P(1)—Os(1)—C(1)	87.5 (15)	C(8)—Os(3)—C(9)	97.9 (19)
P(1)—Os(1)—C(2)	101.8 (13)	C(8)—Os(3)—C(10)	95.7 (18)
P(1)—Os(1)—C(3)	89.2 (14)	C(9)—Os(3)—C(10)	165.1 (16)
C(1)—Os(1)—C(2)	91.4 (19)	Os(1)—P(1)—C(11)	118.3 (17)
C(1)—Os(1)—C(3)	172 (2)	Os(1)—P(1)—C(12)	126 (2)
C(2)—Os(1)—C(3)	96.1 (18)	C(11)—P(1)—C(12)	113 (3)
Os(1)—Os(2)—Os(3)	59.09 (6)	Os(1)—P(1)—C(11′)	121 (3)
Os(1)—Os(2)—C(4)	143.0 (14)	Os(1)—P(1)—C(12′)	119 (3)
Os(1)—Os(2)—C(5)	112.1 (11)	C(11′)—P(1)—C(12′)	110 (4)
Os(1)—Os(2)—C(6)	99.5 (14)	Os(1)—C(1)—O(1)	174 (4)
Os(3)—Os(2)—C(4)	85.6 (14)	Os(1)—C(2)—O(2)	171 (4)
Os(3)—Os(2)—C(5)	166.6 (11)	Os(1)—C(3)—O(3)	169 (4)
Os(3)—Os(2)—C(6)	92.4 (14)	Os(2)—C(4)—O(4)	171 (4)
C(4)—Os(2)—C(5)	100.3 (18)	Os(2)—C(5)—O(5)	174 (3)
C(4)—Os(2)—C(6)	92 (2)	Os(2)—C(6)—O(6)	166 (4)
C(5)—Os(2)—C(6)	99.4 (18)	Os(3)—C(7)—O(7)	170 (4)
Os(1)—Os(3)—Os(2)	62.99 (6)	Os(3)—C(8)—O(8)	179 (4)
Os(1)—Os(3)—C(7)	168.5 (13)	Os(3)—C(9)—O(9)	164 (3)
Os(1)—Os(3)—C(8)	93.3 (15)	OS(3)—C(10)—O(10)	170 (3)

Compound (2)

Crystal data

$[\mathrm{Os_3(C_2H_6P)H(CO)_{10}}]$
M_r = 912.7
Monoclinic
$P2_1/c$
a = 8.860 (3) Å
b = 16.408 (11) Å
c = 13.779 (11) Å
β = 103.40 (6)°
V = 1949 Å^3
Z = 4
D_x = 3.111 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 16 reflections
θ = 7-9°
μ = 19.67 mm^{-1}
T = 298 K
Lath
0.86 × 0.44 × 0.35 mm
Dark red

Data collection

Stoe Stadi-4 four-circle diffractometer
ω-2θ scans
Absorption correction: semi-empirical
$T_{\min}$ = 0.007, $T_{\max}$ = 0.055
2646 measured reflections
2646 independent reflections

2106 observed reflections [$F > 6\sigma(F)$]
$\theta_{\max}$ = 22.5°
h = −9 → 9
k = 0 → 17
l = 0 → 14
3 standard reflections
frequency: 120 min
intensity variation: 2%

Refinement

Refinement on F
$R = 0.0376$
$wR = 0.0524$
$S = 0.620$
2106 reflections
175 parameters
H atoms not located
$w = 1/[\sigma^2(F) + 0.0071F^2]$
$(\Delta/\sigma)_{\max} = 0.08$
$\Delta\rho_{\max} = 1.19$ e Å^{-3}
$\Delta\rho_{\min} = -2.85$ e Å^{-3}
Atomic scattering factors from Cromer & Mann (1968) or inlaid

Table 3. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters* (Å^2) *for* (2)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ for Os, P and O atoms.

	x	y	z	U_{iso}/U_{eq}
Os(1)	0.14457 (6)	0.0293 (3)	0.31289 (3)	0.0383 (4)
Os(2)	0.32001 (6)	0.05959 (3)	0.16385 (3)	0.0365 (4)
Os(3)	0.11790 (6)	0.18474 (3)	0.21506 (4)	0.0404 (4)
P(1)	0.3913 (4)	0.18638 (19)	0.2419 (3)	0.048 (2)
C(11)	−0.0176 (18)	−0.0075 (9)	0.1969 (11)	0.055 (3)
C(12)	0.206 (2)	−0.0816 (11)	0.3517 (12)	0.066 (4)
C(13)	0.3052 (19)	0.0664 (9)	0.4214 (12)	0.061 (4)
C(14)	−0.0152 (16)	0.0494 (7)	0.3841 (10)	0.047 (3)
C(21)	0.222 (2)	−0.0386 (10)	0.0978 (13)	0.069 (4)
C(22)	0.4942 (16)	0.0025 (8)	0.2307 (10)	0.050 (3)
C(23)	0.3996 (16)	0.0875 (8)	0.0527 (11)	0.050 (3)
C(31)	−0.098 (2)	0.1594 (10)	0.1737 (12)	0.071 (4)
C(32)	0.1060 (18)	0.2369 (9)	0.3337 (11)	0.057 (4)
C(33)	0.1101 (19)	0.2817 (10)	0.1388 (12)	0.065 (4)
C(1*p*)	0.462 (2)	0.2617 (10)	0.1618 (12)	0.070 (4)
C(2*p*)	0.530 (3)	0.2008 (11)	0.3627 (15)	0.087 (5)
O(11)	−0.1097 (13)	−0.0320 (7)	0.1386 (8)	0.080 (8)
O(12)	0.2408 (16)	−0.1469 (7)	0.3723 (12)	0.101 (10)
O(13)	0.393 (2)	0.0856 (9)	0.4929 (9)	0.109 (11)
O(14)	−0.1111 (12)	0.0617 (7)	0.4237 (8)	0.075 (7)
O(21)	0.1745 (17)	−0.0978 (8)	0.0585 (10)	0.105 (10)
O(22)	0.6062 (15)	−0.0307 (7)	0.2745 (10)	0.087 (9)
O(23)	0.4471 (15)	0.1036 (7)	−0.0167 (9)	0.086 (9)
O(31)	−0.2298 (15)	0.1478 (8)	0.1467 (11)	0.099 (10)
O(32)	0.103 (3)	0.2737 (7)	0.4025 (10)	0.121 (12)
O(33)	0.1015 (15)	0.3400 (7)	0.0894 (10)	0.0961 (9)

Table 4. *Geometric parameters* (Å, °) *for* (2)

Os(1)—Os(2)	2.8916 (7)	Os(3)—C(31)	1.913 (18)
Os(1)—Os(3)	2.8698 (7)	Os(3)—C(32)	1.870 (15)
Os(2)—Os(3)	2.9166 (7)	Os(3)—C(33)	1.899 (16)
Os(2)—P(1)	2.360 (4)	C(11)—O(11)	1.080 (19)
Os(3)—P(1)	2.365 (4)	C(12)—O(12)	1.13 (2)
P(1)—C(1*p*)	1.858 (17)	C(13)—O(13)	1.15 (2)
P(1)—C(2*p*)	1.84 (2)	C(14)—O(14)	1.129 (17)
Os(1)—C(11)	1.980 (15)	C(21)—O(21)	1.14 (2)
Os(1)—C(12)	1.938 (17)	C(22)—O(22)	1.170 (19)
Os(1)—C(13)	1.909 (16)	C(23)—O(23)	1.161 (19)
Os(1)—C(14)	1.929 (14)	C(31)—O(31)	1.16 (2)
Os(2)—C(21)	1.954 (18)	C(32)—O(32)	1.13 (2)
Os(2)—C(22)	1.857 (14)	C(33)—O(33)	1.17 (2)
Os(2)—C(23)	1.884 (14)		
Os(1)—Os(2)—Os(3)	59.219 (17)	C(12)—Os(1)—C(14)	102.2 (7)
Os(1)—Os(3)—Os(2)	59.955 (17)	C(13)—Os(1)—C(14)	92.8 (6)
Os(2)—Os(1)—Os(3)	60.826 (17)	Os(1)—Os(2)—C(21)	86.1 (5)
Os(1)—Os(2)—P(1)	87.68 (9)	Os(1)—Os(2)—C(22)	94.7 (4)
Os(3)—Os(2)—P(1)	51.96 (9)	Os(1)—Os(2)—C(23)	169.4 (4)
Os(1)—Os(3)—P(1)	88.10 (9)	Os(3)—Os(2)—C(21)	117.4 (5)
Os(2)—Os(3)—P(1)	51.80 (9)	Os(3)—Os(2)—C(22)	136.2 (4)
Os(2)—P(1)—Os(3)	76.24 (11)	Os(3)—Os(2)—C(23)	113.1 (4)
Os(2)—P(1)—C(1*p*)	114.0 (5)	C(21)—Os(2)—C(22)	92.6 (7)
Os(2)—P(1)—C(2*p*)	125.3 (7)	C(21)—Os(2)—C(23)	91.9 (7)
Os(3)—P(1)—C(1*p*)	112.8 (5)	C(22)—Os(2)—C(23)	95.8 (6)
Os(3)—P(1)—C(2*p*)	125.9 (7)	Os(1)—Os(3)—C(31)	85.1 (5)
C(1P)—P(1)—C(2*p*)	102.0 (8)	Os(1)—Os(3)—C(32)	90.7 (5)
P(1)—Os(2)—C(21)	169.3 (5)	Os(1)—Os(3)—C(33)	173.3 (5)
P(1)—Os(2)—C(22)	96.6 (4)	Os(2)—Os(3)—C(31)	113.8 (5)
P(1)—Os(2)—C(23)	92.6 (4)	Os(2)—Os(3)—C(32)	134.1 (5)
P(1)—Os(3)—C(31)	165.4 (5)	Os(2)—Os(3)—C(33)	113.7 (5)
P(1)—Os(3)—C(32)	97.0 (5)	C(31)—Os(3)—C(32)	96.0 (7)
P(1)—Os(3)—C(33)	88.9 (5)	C(31)—Os(3)—C(33)	96.5 (7)
Os(2)—Os(1)—C(11)	83.6 (4)	C(32)—Os(3)—C(33)	95.6 (7)
Os(2)—Os(1)—C(12)	101.3 (5)	Os(1)—C(11)—O(11)	174.1 (14)
Os(2)—Os(1)—C(13)	94.3 (5)	Os(1)—C(12)—O(12)	178.6 (16)
Os(2)—Os(1)—C(14)	155.6 (4)	Os(1)—C(13)—O(13)	173.2 (15)
Os(3)—Os(1)—C(11)	85.8 (4)	Os(1)—C(14)—O(14)	178.4 (12)
Os(3)—Os(1)—C(12)	162.1 (5)	Os(2)—C(21)—O(21)	174.9 (16)
Os(3)—Os(1)—C(13)	92.8 (5)	Os(2)—C(22)—O(22)	177.5 (13)
Os(3)—Os(1)—C(14)	95.5 (4)	Os(2)—C(23)—O(23)	178.8 (13)
C(11)—Os(1)—C(12)	92.4 (7)	Os(3)—C(31)—O(31)	176.7 (16)
C(11)—Os(1)—C(13)	177.8 (7)	Os(3)—C(32)—O(32)	174.6 (15)
C(11)—Os(1)—C(14)	89.0 (6)	Os(3)—C(33)—O(33)	177.6 (15)
C(12)—Os(1)—C(13)	88.5 (7)		

Compound (3)

Crystal data

$[Os_3(CH_3P)(H)_2(CO)_9]$
$M_r = 870.7$
Orthorhombic
Pcab
$a = 17.108$ (4) Å
$b = 14.229$ (4) Å
$c = 14.485$ (9) Å
$V = 3526$ Å^3
$Z = 8$
$D_x = 3.280$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 18 reflections
$\theta = 3\text{-}21°$
$\mu = 21.73$ mm^{-1}
$T = 298$ K
Sphenoid
0.38 × 0.32 × 0.04 mm
Dark red

Data collection

Stoe Stadi-2 two-circle diffractometer
ω scans
Absorption correction: empirical
$T_{\min} = 0.010$, $T_{\max} = 0.125$
3296 measured reflections
3296 independent reflections
1989 observed reflections $[F > 6\sigma(F)]$
$\theta_{\max} = 25°$
$h = 0 \rightarrow 20$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 16$
2 standard reflections
frequency: 720 min
intensity variation: none

Refinement

Refinement on F
$R = 0.0430$
$wR = 0.0526$
$S = 1.076$
1989 reflections
113 parameters
H atoms not located
$w = 1/[\sigma^2(F) + 0.0008F^2]$
$(\Delta/\sigma)_{\max} = 0.02$
$\Delta\rho_{\max} = 1.38$ e Å^{-3}
$\Delta\rho_{\min} = -1.46$ e Å^{-3}
Atomic scattering factors from Cromer & Mann (1968) or inlaid

Table 5. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters* (Å^2) *for* (3)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ for Os and P atoms.

	x	y	z	U_{iso}/U_{eq}
Os(1)	0.20009 (4)	0.3583 (6)	0.09192 (5)	0.0456 (4)
Os(2)	0.07061 (4)	0.23264 (6)	0.03212 (5)	0.0429 (4)
Os(3)	0.04690 (4)	0.35109 (6)	0.18870 (5)	0.0455 (4)
P(1)	0.1393 (3)	0.2336 (4)	0.1700 (4)	0.049 (3)
C(11)	0.2600 (12)	0.3979 (18)	0.1928 (16)	0.069 (6)
C(12)	0.2175 (13)	0.4668 (20)	0.0158 (18)	0.074 (7)
C(13)	0.2825 (13)	0.2839 (18)	0.0474 (16)	0.069 (6)
C(21)	−0.0060 (11)	0.1412 (15)	0.0690 (14)	0.051 (5)
C(22)	0.0022 (12)	0.3003 (17)	−0.0493 (16)	0.066 (6)
C(23)	0.1193 (11)	0.1534 (16)	−0.0524 (14)	0.056 (5)
C(31)	−0.0344 (10)	0.2795 (15)	0.2301 (14)	0.054 (5)

C(32)	−0.0218 (12)	0.4371 (18)	0.1349 (17)	0.071 (6)
C(33)	0.0679 (12)	0.4047 (19)	0.3034 (17)	0.075 (7)
C(1*p*)	0.1855 (12)	0.1464 (18)	0.2451 (18)	0.076 (6)
O(11)	0.2938 (9)	0.4291 (14)	0.2571 (13)	0.094 (5)
O(12)	0.2299 (10)	0.5268 (14)	−0.0338 (13)	0.094 (5)
O(13)	0.3335 (10)	0.2404 (14)	0.0169 (13)	0.095 (6)
O(21)	−0.0521 (9)	0.0903 (14)	0.0874 (12)	0.085 (5)
O(22)	−0.0411 (9)	0.3444 (12)	−0.0931 (12)	0.080 (5)
O(23)	0.1494 (9)	0.1025 (13)	−0.1042 (12)	0.084 (5)
O(31)	−0.0902 (9)	0.2345 (14)	0.2526 (13)	0.095 (5)
O(32)	−0.0628 (10)	0.4918 (15)	0.0946 (14)	0.102 (6)
O(33)	0.0874 (10)	0.4390 (14)	0.3750 (14)	0.097 (5)

Table 6. *Geometric parameters* (Å, °) *for* (3)

Os(1)—Os(2)	2.9776 (11)	Os(3)—C(31)	1.83 (2)
Os(1)—Os(3)	2.9741 (11)	Os(3)—C(32)	1.87 (2)
Os(2)—Os(3)	2.8547 (11)	Os(3)—C(33)	1.86 (3)
Os(1)—P(1)	2.350 (5)	C(11)—O(11)	1.18 (3)
Os(2)—P(1)	2.318 (5)	C(12)—O(12)	1.14 (3)
Os(3)—P(1)	2.317 (5)	C(13)—O(13)	1.16 (3)
P(1)—C(1*p*)	1.83 (3)	C(21)—O(21)	1.10 (3)
Os(1)—C(11)	1.87 (2)	C(22)—O(22)	1.16 (3)
Os(1)—C(12)	1.92 (3)	C(23)—O(23)	1.16 (3)
Os(1)—C(13)	1.88 (2)	C(31)—O(31)	1.19 (3)
Os(2)—C(21)	1.92 (2)	C(32)—O(32)	1.20 (3)
Os(2)—C(22)	1.92 (2)	C(33)—O(33)	1.19 (3)
Os(2)—C(23)	1.86 (2)		
Os(2)—Os(1)—Os(3)	57.33 (3)	Os(1)—Os(2)—C(21)	146.5 (6)
Os(1)—Os(2)—Os(3)	61.28 (3)	Os(1)—Os(2)—C(22)	109.3 (7)
Os(1)—Os(3)—Os(2)	61.40 (3)	Os(1)—Os(2)—C(23)	102.9 (6)
Os(2)—Os(1)—P(1)	49.89 (13)	Os(3)—Os(2)—C(21)	94.7 (6)
Os(3)—Os(1)—P(1)	49.92 (13)	Os(3)—Os(2)—C(22)	96.0 (7)
Os(1)—Os(2)—P(1)	50.84 (13)	Os(3)—Os(2)—C(23)	160.7 (6)
Os(3)—Os(2)—P(1)	51.96 (13)	Os(1)—Os(3)—C(31)	147.8 (6)
Os(1)—Os(3)—P(1)	50.90 (13)	Os(1)—Os(3)—C(32)	109.5 (7)
Os(2)—Os(3)—P(1)	52.00 (13)	Os(1)—Os(3)—C(33)	103.70 (8)
Os(1)—P(1)—Os(2)	79.27 (17)	Os(2)—Os(3)—C(31)	92.3 (6)
Os(1)—P(1)—Os(3)	79.18 (17)	Os(2)—Os(3)—C(32)	98.3 (7)
Os(1)—P(1)—C(1*p*)	127.5 (8)	Os(2)—Os(3)—C(33)	157.3 (8)
Os(2)—P(1)—Os(3)	76.04 (16)	C(11)—Os(1)—C(12)	97.1 (10)
Os(2)—P(1)—C(1*p*)	136.7 (8)	C(11)—Os(1)—C(13)	91.5 (10)
Os(3)—P(1)—C(1*p*)	135.6 (8)	C(12)—Os(1)—C(13)	98.0 (10)
P(1)—Os(1)—C(11)	95.3 (7)	C(21)—Os(2)—C(22)	95.4 (9)
P(1)—Os(1)—C(12)	162.4 (8)	C(21)—Os(2)—C(23)	94.5 (9)
P(1)—Os(1)—C(13)	94.0 (7)	C(22)—Os(2)—C(23)	100.0 (9)
P(1)—Os(2)—C(22)	146.7 (7)	C(31)—Os(3)—C(32)	91.4 (10)
P(1)—Os(2)—C(23)	110.1 (7)	C(31)—Os(3)—C(33)	94.7 (10)
P(1)—Os(2)—C(21)	96.3 (6)	C(32)—Os(3)—C(33)	103.0 (11)
P(1)—Os(3)—C(31)	98.9 (6)	Os(1)—C(11)—O(11)	175 (2)
P(1)—Os(3)—C(32)	148.6 (7)	Os(1)—C(12)—O(12)	175 (2)
P(1)—Os(3)—C(33)	105.5 (8)	Os(1)—C(13)—O(13)	177 (2)
Os(2)—Os(1)—C(11)	144.6 (7)	Os(2)—C(21)—O(21)	177.0 (19)
Os(2)—Os(1)—C(12)	115.6 (8)	Os(2)—C(22)—O(22)	175 (2)
Os(2)—Os(1)—C(13)	96.8 (7)	Os(2)—C(23)—O(23)	178.8 (19)
Os(3)—Os(1)—C(11)	97.2 (7)	Os(3)—C(31)—O(31)	175.9 (18)
Os(3)—Os(1)—C(12)	115.9 (8)	Os(3)—C(32)—O(32)	175 (2)
Os(3)—Os(1)—C(13)	143.4 (7)	Os(3)—C(33)—O(33)	175 (2)

The structure of (1) was solved by direct methods using *SHELXS*84 (Sheldrick, 1984). The methyl groups of the phosphine were each found to be disordered over two sites, such that the occupancy of C(11) and C(12) refined to 0.606 (9) and that of the alternative positions represented by C(11′) and C(12′) to 0.394 (9). Only the Os atoms were allowed anisotropic thermal motion. The absolute structure was determined and is presented here; the other polarity gave higher residuals *R* and *wR* of 0.0450 and 0.0493, respectively, and higher errors in atomic positional and displacement parameters. The structures of compounds (2) and (3) were solved similarly. The atoms allowed anisotropic thermal motion were Os, P and O for (2) and Os and P for (3). The programs used were *SHELX*76 (Sheldrick, 1976) and *SHELXS*86 (Sheldrick, 1985). *CALC* (Gould & Taylor, 1985) was used for molecular geometry calculations.

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Lists of structure factors and anisotropic displacement parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71506 (36 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HU1044]

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Tetra-*n*-butylammonium Tris(perfluorophenyl)(triphenylphosphine)platinate(II)

IRENE ARA AND MILAGROS TOMÁS*

Departamento de Química Inorgánica,
Instituto de Ciencia de Materiales de Aragón,
Univesidad de Zaragoza-CSIC, 50009 Zaragoza, Spain

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Abstract

The coordination geometry of the ligands around the Pt atom is approximately square planar in $[N(C_4H_9)_4][Pt(C_6F_5)_3(C_{18}H_{15}P)]$, with Pt—C dis-