sites; this was allowed for in the refinement and the value of the P—F bond length obtained is 1·549 (4) Å, in accord with accepted values. Chemical studies on the other crystalline material mentioned in the *Introduction* are in progress (Senoff, 1978).

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μ4-Benzylidyne-μ3-benzylidyne-hexadecacarbonylhexaosmium

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Abstract. $Os_6(CO)_{16}[C(C_6H_5)]_2$, monoclinic, $P2_1/c$, $a = 22\cdot813$ (10), $b = 10\cdot603$ (5), $c = 29\cdot003$ (10) Å, $\beta = 91\cdot55$ (3)°, $U = 7012\cdot8$ Å^3, $Z = 8$, $D_c = 3\cdot35$ g cm^{-3}, $\mu(Mo\ K\alpha) = 217\cdot24$ cm^{-1}. The structure was refined to an R of 0·062 for 5811 unique diffractometer data. The two independent molecules are structurally equivalent; the six Os atoms, in each cluster, define a monocapped square-based pyramid, with one benzylidyne ligand capping the square face, and the other an adjacent triangular face. The carbonyl ligands are all terminal.

Introduction. $Os_6(CO)_{16}[C(C_6H_5)]_2$ was isolated as one of the products of the photolysis of $Os_6(CO)_{18}$ with phenylacetylene (Fernandez, Johnson & Lewis, 1977). Spectroscopic data showed the compound to be an organometallic cluster based on the Os_6 unit, but neither the geometry of the metal cluster nor the nature of the organic species could be established. A single-crystal X-ray analysis was therefore undertaken.

Crystals of $Os_6(CO)_{16}[C(C_6H_5)]_2$ were obtained as dark-red tablets from an ethyl acetate/hexane mixture. 8193 intensities were measured (for $3\cdot0 < 2\theta \leq 60\cdot0°$) on a Syntex $P2_1$ four-circle diffractometer, using graphite-monochromated Mo $K\alpha$ radiation, a θ–2θ scan procedure, and a crystal with dimensions 0·315 × 0·175 × 0·105 mm. Lp corrections and semi-empirical absorption corrections (based on a pseudo-ellipsoid model and 460 azimuthal scan data from 44 independent reflections which gave transmission coefficients ranging from 0·665 to 0·206 for the full data set) were applied. The data were averaged to give 5811 unique observed reflections [$I > 1\cdot5\sigma(I)$]. Cell dimensions were derived from the angular measurements of 15 strong reflections ($20\cdot0 < 2\theta < 30\cdot0°$).

The Os atoms were located by multisolution $\sum_2$ sign expansion, and the remaining non-hydrogen atoms by difference syntheses. The structure was refined by blocked-full-matrix least squares with anisotropic Os and isotropic light atoms. In view of the relatively large uncertainties in the light-atom positions, the X-ray scattering being dominated by the Os contributions, the Os—C and C—O bond lengths in each molecule were constrained to be equal; these distances were refined [molecule 1: Os—C 1·89 (1), C—O 1·16 (1) Å; molecule 2: Os—C 1·86 (1), C—O 1·18 (1) Å]. The phenyl rings were refined as rigid groups with idealized geometry (C—C 1·395 Å, C—C—C 120·0°). Complex neutral-atom scattering factors (*International Tables for X-ray Crystallography*, 1974), and a weighting scheme of the form $w = 1\cdot384/[\sigma^2(F) + 0\cdot001|F_o|^2]$ were employed. The refinement converged to $R = 0\cdot062$ and $R' = \sum w^{1/2}\Delta/\sum w^{1/2}|F_o| = 0\cdot059$. The final

Table 1. *Atomic coordinates* ($\times 10^4$)

	Molecule 1			Molecule 2		
	x	*y*	*z*	*x*	*y*	*z*
Os(1)	4528 (1)	4571 (1)	1023 (1)	552 (1)	95 (1)	3998 (1)
Os(2)	3430 (1)	5559 (1)	854 (1)	1661 (1)	1068 (1)	4052 (1)
Os(3)	3454 (1)	6732 (1)	1672 (1)	1521 (1)	2165 (1)	3227 (1)
Os(4)	4546 (1)	5803 (1)	1882 (1)	416 (1)	1176 (1)	3111 (1)
Os(5)	3603 (1)	4076 (1)	1657 (1)	1386 (1)	−502 (1)	3295 (1)
Os(6)	2489 (1)	5338 (1)	1414 (1)	2516 (1)	809 (1)	3410 (1)
C(11)	4250 (14)	3358 (23)	593 (10)	893 (18)	−1066 (29)	4408 (12)
O(11)	4115 (12)	2630 (20)	315 (8)	1102 (13)	−1867 (21)	4646 (10)
C(12)	5125 (14)	3401 (31)	1182 (17)	−120 (15)	−874 (44)	3899 (22)
O(12)	5452 (12)	2642 (22)	1312 (12)	−566 (11)	−1449 (30)	3897 (15)
C(13)	5096 (13)	5162 (32)	613 (11)	161 (18)	885 (38)	4471 (11)
O(13)	5448 (10)	5522 (26)	367 (9)	15 (16)	1365 (31)	4818 (8)
C(21)	3022 (15)	4359 (25)	482 (12)	2103 (16)	−99 (27)	4394 (13)
O(21)	2798 (12)	3545 (19)	278 (10)	2359 (13)	−840 (23)	4630 (10)
C(22)	3611 (17)	6329 (32)	285 (8)	1543 (18)	1817 (33)	4621 (8)
O(22)	3687 (13)	6749 (24)	−77 (6)	1476 (13)	2330 (23)	4976 (7)
C(31)	3195 (18)	7006 (38)	2278 (7)	1755 (18)	2279 (37)	2619 (7)
O(31)	3016 (14)	7002 (29)	2647 (7)	1893 (14)	2363 (29)	2229 (6)
C(32)	3636 (14)	8472 (14)	1708 (12)	1395 (15)	3898 (14)	3206 (13)
O(32)	3708 (13)	9528 (15)	1781 (11)	1201 (12)	4918 (17)	3136 (11)
C(41)	4354 (17)	5771 (33)	2514 (6)	−68 (19)	2582 (31)	3047 (21)
O(41)	4193 (12)	5716 (25)	2887 (6)	−380 (14)	3466 (24)	2992 (15)
C(42)	5276 (11)	5024 (37)	2013 (18)	−257 (14)	197 (39)	3034 (20)
O(42)	5714 (9)	4538 (25)	2112 (12)	−643 (13)	−489 (26)	2915 (14)
C(43)	4932 (18)	7361 (22)	1981 (17)	561 (16)	1196 (32)	2482 (6)
O(43)	5126 (12)	8337 (17)	2076 (11)	708 (14)	1158 (29)	2095 (6)
C(51)	4201 (11)	2969 (23)	1876 (12)	759 (15)	−1589 (35)	3174 (19)
O(51)	4544 (11)	2259 (22)	2020 (11)	437 (12)	−2444 (20)	3088 (11)
C(52)	3304 (19)	2659 (26)	1329 (14)	1737 (16)	−1860 (24)	3603 (13)
O(52)	3177 (12)	1742 (17)	1134 (9)	1941 (13)	−2723 (20)	3809 (10)
C(53)	3266 (16)	3913 (35)	2243 (7)	1570 (19)	−875 (38)	2690 (7)
O(53)	3061 (14)	3790 (29)	2601 (7)	1739 (14)	−1036 (30)	2311 (7)
C(61)	1792 (11)	6150 (30)	1204 (15)	3220 (11)	1662 (34)	3511 (17)
O(61)	1362 (10)	6567 (28)	1046 (13)	3673 (8)	2205 (23)	3535 (11)
C(62)	2096 (16)	3837 (22)	1237 (15)	2757 (19)	606 (39)	2808 (7)
O(62)	1909 (13)	2884 (18)	1109 (11)	2893 (16)	444 (33)	2422 (7)
C(63)	2175 (23)	5430 (50)	2010 (9)	2881 (20)	−622 (28)	3659 (18)
O(63)	1980 (15)	5414 (33)	2374 (7)	3197 (13)	−1436 (25)	3795 (13)
C(201)	4196 (15)	6474 (28)	1218 (13)	847 (13)	1941 (25)	3740 (11)
C(202)	4452 (10)	7647 (16)	987 (8)	594 (10)	3101 (17)	3981 (9)
C(203)	4111 (10)	8589 (16)	778 (8)	976 (10)	4040 (17)	4141 (9)
C(204)	4379 (10)	9649 (16)	592 (8)	756 (10)	5113 (17)	4354 (9)
C(205)	4989 (10)	9767 (16)	616 (8)	153 (10)	5246 (17)	4408 (9)
C(206)	5329 (10)	8825 (16)	825 (8)	−230 (10)	4307 (17)	4247 (9)
C(207)	5061 (10)	7765 (16)	1011 (8)	−9 (10)	3235 (17)	4034 (9)
C(211)	2907 (14)	6979 (26)	1123 (12)	2140 (16)	2442 (31)	3736 (14)
C(212)	2566 (10)	8013 (18)	923 (9)	2487 (11)	3520 (20)	3922 (9)
C(213)	2336 (10)	8900 (18)	1224 (9)	2629 (11)	4505 (20)	3627 (9)
C(214)	1985 (10)	9880 (18)	1052 (9)	3002 (11)	5468 (20)	3781 (9)
C(215)	1865 (10)	9971 (18)	579 (9)	3233 (11)	5446 (20)	4231 (9)
C(216)	2094 (10)	9083 (18)	278 (9)	3091 (11)	4461 (20)	4527 (9)
C(217)	2445 (10)	8104 (18)	450 (9)	2718 (11)	3498 (20)	4373 (9)

atomic coordinates are given in Table 1, while some important bond lengths and angles are listed in Tables 2 and 3 respectively.*

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* Lists of structure factors, thermal parameters and bond angles involving the ligands have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33672 (40 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Discussion. $Os_6(CO)_{16}[C(C_6H_5)]_2$ crystallizes with two structurally equivalent molecules in the asymmetric unit (molecule 1 is depicted in Fig. 1). The geometry of the metal framework is similar to that found in $H_2Os_6(CO)_{18}$ (McPartlin, Eady, Johnson & Lewis, 1976). The structure may be derived from the dihydride by the replacement of the two H atoms, and two carbonyls (one from each Os atom in the basal plane, which is bonded to the face-capping metal) by the two

$C(C_6H_5)$ groups. Each organic species is considered to act as a three-electron donor, so that both compounds are 86-electron systems.

The apical metal atom, Os(5), lies 2·10 Å above the Os_4 basal plane, and is displaced slightly towards the Os(2)—Os(3) edge. The Os_4 base shows some bond-length variation, the shortest edge, Os(2)—Os(3), being the one bridged by Os(6). This face-capping metal forms relatively short bonds to the two basal atoms, Os(2) and Os(3), but the bond to the apical metal, Os(5), is the longest in the cluster. This may be of energetic significance, since it is this long bond which ruptures upon the inclusion of an additional electron pair (Eady, Fernandez, Johnson, Lewis, Raithby & Sheldrick, 1978).

Table 2. *Selected bond lengths* (Å)

	Molecule 1	Molecule 2
Os(2)—Os(1)	2·747 (3)	2·733 (3)
Os(4)—Os(1)	2·813 (3)	2·826 (3)
Os(5)—Os(1)	2·882 (3)	2·894 (3)
Os(3)—Os(2)	2·678 (3)	2·672 (3)
Os(5)—Os(2)	2·827 (3)	2·812 (3)
Os(6)—Os(2)	2·736 (3)	2·746 (3)
Os(4)—Os(3)	2·731 (3)	2·742 (3)
Os(5)—Os(3)	2·837 (3)	2·852 (3)
Os(6)—Os(3)	2·739 (3)	2·728 (3)
Os(5)—Os(4)	2·886 (3)	2·879 (3)
Os(6)—Os(5)	2·941 (3)	2·939 (3)
C(201)—Os(1)	2·233 (32)	2·206 (28)
C(201)—Os(2)	2·239 (35)	2·245 (30)
C(201)—Os(3)	2·189 (37)	2·181 (33)
C(201)—Os(4)	2·185 (37)	2·204 (32)
C(211)—Os(2)	2·084 (31)	2·053 (37)
C(211)—Os(3)	2·015 (33)	2·035 (40)
C(211)—Os(6)	2·168 (31)	2·161 (36)
C(202)—C(201)	1·535 (38)	1·535 (36)
C(212)—C(211)	1·455 (37)	1·484 (42)
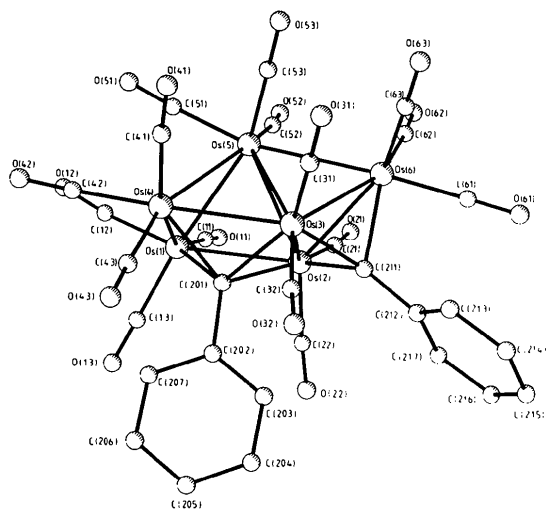
Fig. 1. Molecule 1 with the atom-numbering scheme.

Table 3. *Selected bond angles* (°)

	Molecule 1	Molecule 2
Os(4)—Os(1)—Os(2)	88·4 (1)	88·8 (1)
Os(5)—Os(1)—Os(2)	60·3 (1)	59·9 (1)
Os(5)—Os(1)—Os(4)	60·9 (1)	60·4 (1)
Os(3)—Os(2)—Os(1)	91·2 (1)	91·4 (1)
Os(5)—Os(2)—Os(1)	62·2 (1)	62·9 (1)
Os(5)—Os(2)—Os(3)	62·0 (1)	62·6 (1)
Os(6)—Os(2)—Os(1)	126·0 (1)	126·6 (1)
Os(6)—Os(2)—Os(3)	60·8 (1)	60·4 (1)
Os(6)—Os(2)—Os(5)	63·8 (1)	63·8 (1)
Os(4)—Os(3)—Os(2)	91·6 (1)	91·8 (1)
Os(5)—Os(3)—Os(2)	61·6 (1)	61·1 (1)
Os(5)—Os(3)—Os(4)	62·4 (1)	61·9 (1)
Os(6)—Os(3)—Os(2)	60·7 (1)	61·1 (1)
Os(6)—Os(3)—Os(4)	126·0 (1)	125·4 (1)
Os(6)—Os(3)—Os(5)	63·6 (1)	63·5 (1)
Os(3)—Os(4)—Os(1)	88·7 (1)	88·0 (1)
Os(5)—Os(4)—Os(3)	60·7 (1)	61·0 (1)
Os(5)—Os(4)—Os(3)	60·6 (1)	60·9 (1)
Os(2)—Os(5)—Os(1)	57·5 (1)	57·2 (1)
Os(3)—Os(5)—Os(1)	85·4 (1)	84·6 (1)
Os(3)—Os(5)—Os(2)	56·4 (1)	56·3 (1)
Os(4)—Os(5)—Os(1)	58·4 (1)	58·6 (1)
Os(4)—Os(5)—Os(2)	85·5 (1)	86·2 (1)
Os(4)—Os(5)—Os(3)	57·0 (1)	57·2 (1)
Os(6)—Os(5)—Os(1)	114·0 (1)	114·1 (1)
Os(6)—Os(5)—Os(2)	56·6 (1)	57·0 (1)
Os(6)—Os(5)—Os(3)	56·6 (1)	56·2 (1)
Os(6)—Os(5)—Os(4)	113·5 (1)	113·4 (1)
Os(3)—Os(6)—Os(2)	58·6 (1)	58·4 (1)
Os(5)—Os(6)—Os(2)	59·6 (1)	59·2 (1)
Os(5)—Os(6)—Os(3)	59·8 (1)	60·3 (1)
Os(2)—C(201)—Os(1)	75·8 (10)	75·8 (9)
Os(3)—C(201)—Os(1)	122·5 (14)	123·7 (13)
Os(3)—C(201)—Os(2)	74·4 (11)	74·3 (9)
Os(4)—C(201)—Os(1)	79·1 (10)	79·7 (9)
Os(4)—C(201)—Os(2)	122·4 (15)	121·9 (13)
Os(4)—C(201)—Os(3)	77·3 (12)	77·4 (11)
Os(3)—C(211)—Os(2)	81·5 (11)	81·6 (13)
Os(6)—C(211)—Os(2)	80·1 (10)	81·3 (12)
Os(6)—C(211)—Os(3)	81·7 (11)	81·0 (13)

The μ_4-benzylidyne ligand symmetrically caps the square basal face, with C(201) lying 1·06 Å below it. The μ_3-benzylidyne caps the triangular face asymmetrically, being displaced towards the Os(2)—Os(3) edge, with the bridging atom 1·37 Å below the plane. The C—C(phenyl) bond vectors are approximately perpendicular to the cluster faces. Similar modes of coordination have been reported for the ethylidyne ligands in $Os_6(CO)_{16}[C(CH_3)]_2$ (Eady *et al.*, 1978), which is derived from the benzylidyne complex by the replacement of the phenyl rings with methyl groups.

The carbonyl ligands are all approximately linear (mean Os—C—O angle = 173·9°), and the Os—C and C—O distances are in agreement with mean values of 1·89 and 1·14 Å in $Os_6(CO)_{18}$ (Mason, Thomas & Mingos, 1973).

bridge University IBM 370/165 computer and programs written by Dr G. M. Sheldrick; the figure was drawn with *PLUTO*, written by Dr W. D. S. Motherwell.

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Racemic 3-[Benzyl(phenyl)phosphinyl]-2-butenoic Acid

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Abstract. $C_{17}H_{17}O_3P$, $M_r = 300.3$, monoclinic, $P2_1/c$, $Z = 4$, $a = 9.206$ (3), $b = 10.530$ (4), $c = 17.223$ (5) Å, $\beta = 111.89$ (6)°, $V = 1549.2$ (Å)^3, $D_x = 1.287$, $D_m = 1.282$ g cm^{-3} (by flotation in a mixture of benzene and bromobenzene), $F(000) = 632$. The structure was solved with *MULTAN* and refined by full-matrix least-squares calculations to a final R of 0.066 ($R_w = 0.050$) for 2078 reflections measured on a Syntex diffractometer. Molecules with the same configuration form chains by means of strong hydrogen bonds of the type

$$\geq P=O\cdots H-O-\underset{\underset{O}{\|}}{C}-.$$

2θ mode ($2\theta \leq 114°$). From 2078 recorded reflections, 273 had $F \leq 2.0\sigma(F)$. The intensities were not corrected for absorption ($\mu = 12.6$ cm^{-1}). The structure was solved by a multisolution technique (*MULTAN*, Germain, Main & Woolfson, 1971). The best solution was characterized by ABSFOM = 1.32, RESID = 29.32 and COMFOM = 3.00 for 356 $E \geq 1.4$. The resulting E map revealed all the atoms but one (in the benzene ring) which was placed geometrically. The structure was then refined by full-matrix least-squares calculations minimizing $\sum w(\Delta F)^2$. After four cycles of refinement with isotropic ($R_w = 0.121$) and a

Introduction. The present study is a continuation of an investigation into the structure and hydrogen bonding in phosphine oxide derivatives containing a carboxylic group (Gałdecki & Główka, 1977*a,b*, 1978). It seemed interesting to determine the structure of a compound in which two groups taking part in the hydrogen bonding are separated by a rigid fragment (here the C=C double bond). Although Leiserowitz (1976, and references therein) discussed exhaustively the hydrogen bonding and geometry of α,β-unsaturated carboxylic acids in the crystal form, no analogy should be expected because of the different functional groups participating in the hydrogen bonding and their different positions in the molecule.

Prismatic crystals were obtained by the slow cooling of a saturated solution in a hot mixture of acetone and water. Intensities from a spherical crystal (about 0.3 mm in diameter) were collected on a Syntex $P2_1$ four-circle diffractometer with graphite-monochromated Cu $K\alpha$ radiation. Measurements were carried out in the θ–

Table 1. *Atomic coordinates ($\times 10^4$) of the non-hydrogen atoms*

	x	y	z
P	8636 (2)	7707 (1)	2523 (1)
O(1)	7976 (4)	6398 (4)	2336 (3)
O(2)	4525 (5)	10213 (4)	2724 (3)
O(3)	3066 (5)	8624 (5)	2029 (4)
C(1)	214 (7)	7813 (6)	3523 (4)
C(2)	45 (8)	7100 (8)	4177 (5)
C(3)	1250 (10)	7165 (9)	4994 (5)
C(4)	2568 (10)	7945 (9)	5132 (5)
C(5)	2711 (9)	8625 (8)	4464 (6)
C(6)	1537 (8)	8573 (7)	3660 (5)
C(7)	7207 (7)	8883 (6)	2563 (4)
C(8)	5759 (7)	8423 (6)	2345 (4)
C(9)	4319 (7)	9116 (6)	2339 (4)
C(10)	9418 (7)	8284 (6)	1756 (4)
C(11)	8170 (7)	8191 (7)	881 (4)
C(12)	7396 (8)	9299 (7)	484 (5)
C(13)	6358 (10)	9211 (9)	−352 (5)
C(14)	5930 (9)	8052 (10)	−752 (5)
C(15)	6698 (10)	6939 (9)	−365 (5)
C(16)	7820 (8)	7012 (7)	457 (5)
C(17)	7812 (8)	10219 (6)	2848 (5)