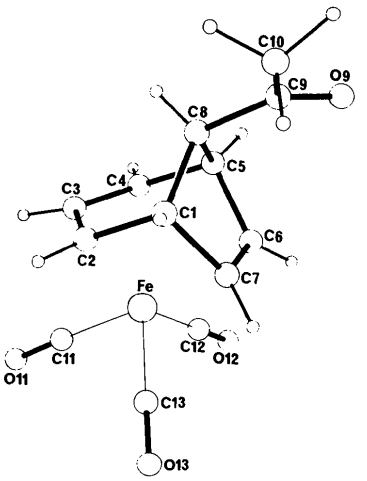

Fig. 1. The $[(C_{10}H_{11}O)Fe(CO)_3]^+$ cation, showing the atom labelling.

consistent with a π-allyl interaction between Fe and C(2)–C(3)–C(4), and a π-alkene interaction between Fe and C(6)–C(7). There is no evidence to support further delocalization in the C_7 ring, as proposed by Margulis, Schiff & Rosenblum (1965). Although C(1), C(7), C(6) and C(5) are coplanar to within 0·004 Å, C(1) and C(5) lie 0·55 and 0·53 Å respectively above the plane defined by C(2), C(3) and C(4). The acetyl group is *endo* to the localized double bond [C(6)–C(7)], with a dihedral angle C(5)–C(8)–C(9)–O(9) of 2·1°. Except for the acetyl group, the cation possesses approximate mirror symmetry through C(3), C(8), Fe, C(11) and O(11). The valence angles at Fe are consistent with this mirror plane but indicate appreciable distortion from local C_{3v} symmetry for the $Fe(CO)_3$ group; however, there are no significant variations in Fe–C (mean 1·807) or C–O (mean 1·132 Å) lengths within this group. The Fe–C–O units are almost linear, with a mean angle of 177·7°.

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References

BROOKHART, M. & DAVIS, E. R. (1971). *Tetrahedron Lett.* pp. 4349–4352.

CHARLES, A. D., DIVERSI, P., JOHNSON, B. F. G., KARLIN, K., LEWIS, J., RIVERA, A. V. & SHELDRICK, G. M. (1977). *J. Organomet. Chem.* **128**, C31–C34.

DAVISON, A., MCFARLANE, W., PRATT, L. & WILKINSON, G. (1962). *J. Chem. Soc.* pp. 4821–4829.

JOHNSON, B. F. G., LEWIS, J. & RANDALL, G. L. P. (1971). *J. Chem. Soc. A*, pp. 422–429.

MARGULIS, T. N., SCHIFF, L. & ROSENBLUM, M. (1965). *J. Am. Chem. Soc.* **87**, 3269–3270.

Acta Cryst. (1978). B**34**, 1718–1722

Bis(triphenylphosphine)iminium μ-Hydrido-decacarbonyltriosmium-μ-carboxylato-heptadecacarbonylhexaosmate, $[(Ph_3P)_2N]^+[HOs_3(CO)_{10}.O_2C.Os_6(CO)_{17}]^-$

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Abstract. $C_{64}H_{31}NO_{29}P_2Os_9$, monoclinic, $P2_1/c$, a = 18·75 (1), b = 15·20 (1), c = 28·41 (1) Å, β = 114·5 (1)°, U = 7368 Å^3, Z = 4, D_x = 2·751 g cm^{-3}. The structure was refined to an R of 0·076 for 3274 unique diffractometer data. The complex anion consists of Os_3 and Os_6 units linked by a CO_2 bridge.

Introduction. The anion was prepared from $Os_6(CO)_{18}$ and $[Os_3(CO)_{11}H]^-$ in CH_2Cl_2, and isolated as the $[(Ph_3P)_2N]^+$ salt (Eady, Guy, Johnson, Lewis, Malatesta & Sheldrick, 1976). The crystal structure determination reveals not an Os_9 cluster as intended, but Os_3 and Os_6 clusters linked by a novel CO_2 bridge.

Dark-brown crystals were obtained by slow diffusion of cyclohexane into a solution in dichloromethane. Intensities were measured with a Syntex $P2_1$ four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation, and a crystal 0·28 × 0·11 × 0·09 mm. 5014 reflexions ($1 \le 2\theta \le 55°$) were measured and corrected for absorption (μ = 150 cm^{-1}); the 3274 data with $F > 4\sigma(F)$ were employed for structure refinement. The nine Os atoms were found by multisolution $\sum_2$ sign

Table 1. *Atom coordinates* ($\times 10^4$) *and isotropic temperature factors* ($Å^2 \times 10^3$) *for the anion*

	x	y	z	U
Os(1)	5427 (2)	2852 (2)	3788 (1)	
Os(2)	4751 (2)	1339 (2)	3151 (1)	
Os(3)	4010 (2)	2111 (2)	3766 (1)	
Os(4)	2350 (2)	2886 (2)	1940 (1)	
Os(5)	1286 (2)	3144 (2)	865 (1)	
Os(6)	2894 (2)	3042 (2)	1137 (1)	
Os(7)	2076 (2)	1546 (2)	1177 (1)	
Os(8)	1833 (2)	2264 (2)	223 (1)	
Os(9)	1976 (2)	4124 (2)	320 (1)	
C(1)	5957 (42)	2083 (47)	4338 (28)	64 (4)
O(1)	6361 (31)	1742 (33)	4721 (21)	83 (3)
C(2)	4832 (43)	3506 (47)	3221 (28)	64 (4)
O(2)	4409 (31)	4014 (34)	2869 (20)	83 (3)
C(3)	6334 (43)	3105 (46)	3643 (26)	64 (4)
O(3)	6857 (30)	3097 (33)	3522 (19)	83 (3)
C(4)	5552 (42)	3825 (48)	4211 (28)	64 (4)
O(4)	5680 (29)	4400 (34)	4526 (20)	83 (3)
C(5)	5458 (44)	679 (48)	3658 (28)	64 (4)
O(5)	5867 (31)	170 (34)	3969 (20)	83 (3)
C(6)	5602 (43)	1529 (47)	2846 (28)	64 (4)
O(6)	5970 (31)	1712 (33)	2666 (20)	83 (3)
C(7)	4328 (42)	415 (49)	2711 (28)	64 (4)
O(7)	4049 (31)	−244 (34)	2472 (19)	83 (3)
C(8)	4536 (41)	1773 (47)	4388 (27)	64 (4)
O(8)	4837 (30)	1150 (33)	4757 (19)	83 (3)
C(9)	3904 (41)	3167 (49)	4087 (28)	64 (4)
O(9)	3910 (30)	3798 (34)	4312 (20)	83 (3)
C(10)	3111 (45)	1641 (46)	3680 (27)	64 (4)
O(10)	2502 (32)	1349 (33)	3678 (19)	83 (3)
C(11)	2420 (42)	4033 (51)	2251 (28)	64 (4)
O(11)	2432 (30)	4715 (36)	2409 (19)	83 (3)
C(12)	1670 (43)	2416 (46)	2273 (28)	64 (4)
O(12)	1336 (30)	2371 (33)	2504 (19)	83 (3)
C(13)	654 (43)	2589 (46)	1168 (27)	64 (4)
O(13)	202 (30)	2243 (34)	1280 (19)	83 (3)
C(14)	526 (43)	3238 (46)	299 (28)	64 (4)
O(14)	−117 (31)	3315 (32)	−95 (20)	83 (3)
C(15)	1192 (44)	4341 (50)	1177 (28)	64 (4)
O(15)	1210 (31)	4975 (35)	1325 (20)	83 (3)
C(16)	3557 (43)	3341 (46)	680 (27)	64 (4)
O(16)	3931 (30)	3176 (33)	472 (20)	83 (3)
C(17)	3752 (43)	2279 (49)	1523 (27)	64 (4)
O(17)	4183 (31)	1713 (34)	1707 (20)	83 (3)
C(18)	3328 (43)	4089 (50)	1581 (28)	64 (4)
O(18)	3560 (30)	4658 (35)	1813 (20)	83 (3)
C(19)	2623 (43)	733 (47)	1112 (27)	64 (4)
O(19)	3192 (30)	182 (33)	1084 (19)	83 (3)
C(20)	1240 (45)	792 (47)	941 (28)	64 (4)
O(20)	556 (32)	449 (33)	756 (20)	83 (3)
C(21)	2369 (43)	1203 (47)	1824 (28)	64 (4)
O(21)	2400 (30)	730 (34)	2193 (20)	83 (3)
C(22)	1009 (44)	1523 (48)	−65 (27)	64 (4)
O(22)	407 (32)	1067 (33)	−302 (20)	83 (3)
C(23)	1471 (41)	2662 (46)	−376 (28)	64 (4)
O(23)	1298 (29)	3020 (33)	−841 (19)	83 (3)
C(24)	2556 (44)	1538 (47)	85 (27)	64 (4)
O(24)	3077 (31)	1075 (33)	93 (19)	83 (3)
C(25)	1213 (45)	4412 (47)	−175 (28)	64 (4)
O(25)	577 (31)	4890 (33)	−480 (19)	83 (3)
C(26)	2270 (42)	5177 (48)	691 (28)	64 (4)
O(26)	2411 (30)	5823 (35)	933 (20)	83 (3)
C(27)	2664 (44)	4585 (47)	−66 (28)	64 (4)
O(27)	3003 (49)	4625 (51)	−281 (31)	83 (3)
C(28)	3243 (32)	2602 (34)	2590 (21)	21 (15)
O(28)	3323 (22)	2726 (24)	3053 (15)	40 (11)
O(29)	3801 (24)	2096 (27)	2566 (15)	47 (12)

expansion, and the C and O atoms from difference syntheses. The structure was refined with complex neutral-atom scattering factors and weighting scheme $w = 1/[\sigma^2(F) + 0{\cdot}001|F_o|^2]$ to $R' = \sum w^{1/2}\Delta/\sum w^{1/2}|F_o| = 0{\cdot}072$ and $R = 0{\cdot}076$. The phenyl groups were refined as rigid groups with idealized geometry (C–C 1·395, C–H 1·08 Å; C–C–C 120, C–C–H 120°); anisotropic temperature factors were employed for Os, common isotropic factors for H, carbonyl C, and carbonyl O respectively, and individual isotropic factors for the remaining atoms. No other assignment of atom types to the CO_2 bridge led to acceptable temperature factors for these three atoms. The final positional and isotropic thermal parameters are given in Tables 1 and 2, bond lengths and angles in Tables 3–5. The labelling of the atoms is shown in Fig. 1 and a stereoscopic view in Fig. 2.*

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33348 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Discussion. The structure of the Os_6 cluster may be regarded as a trigonal bipyramid of five $Os(CO)_3$

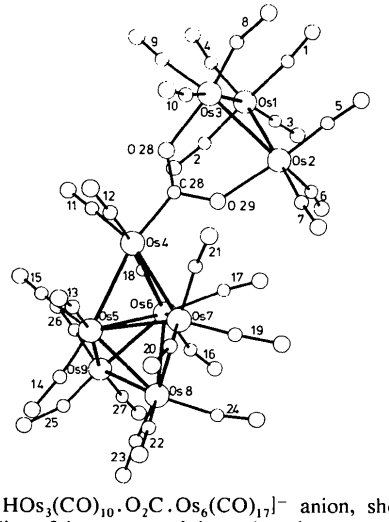

Fig. 1. The $[HOs_3(CO)_{10}.O_2C.Os_6(CO)_{17}]^-$ anion, showing the labelling of the atoms and the carbonyl groups.

Table 2. *Atom coordinates* ($\times 10^4$) *and isotropic thermal parameters* ($Å^2 \times 10^3$) *for the cation*

	x	y	z	U		x	y	z	U
P(1)	8194 (10)	3055 (11)	2001 (6)	38 (5)	C(61)	5528 (28)	1456 (26)	665 (14)	78 (25)
P(2)	7922 (10)	1492 (11)	1323 (6)	36 (5)	C(62)	5378 (28)	1095 (26)	1067 (14)	78 (25)
N	8357 (31)	2152 (35)	1793 (20)	58 (16)	C(63)	5999 (28)	871 (26)	1532 (14)	67 (23)
C(29)	8848 (26)	3887 (21)	1953 (16)	39 (18)	C(64)	6770 (28)	1007 (26)	1597 (14)	72 (24)
C(30)	9551 (26)	3599 (21)	1947 (16)	51 (20)	H(30)	9665	2904	1942	168 (61)
C(31)	10107 (26)	4210 (21)	1948 (16)	63 (22)	H(31)	10651	3988	1943	168 (61)
C(32)	9960 (26)	5109 (21)	1955 (16)	75 (25)	H(32)	10390	5582	1956	168 (61)
C(33)	9257 (26)	5397 (21)	1961 (16)	45 (19)	H(33)	9143	6092	1966	168 (61)
C(34)	8702 (26)	4786 (21)	1960 (16)	19 (15)	H(34)	8158	5008	1965	168 (61)
C(35)	7220 (27)	3505 (25)	1693 (13)	57 (21)	H(36)	6759	2892	2190	168 (61)
C(36)	6621 (27)	3247 (25)	1834 (13)	42 (18)	H(37)	5380	3250	1624	168 (61)
C(37)	5843 (27)	3449 (25)	1515 (13)	62 (22)	H(38)	5062	4065	807	168 (61)
C(38)	5664 (27)	3909 (25)	1055 (13)	75 (25)	H(39)	6125	4522	557	168 (61)
C(39)	6263 (27)	4166 (25)	914 (13)	60 (22)	H(40)	7504	4163	1124	168 (61)
C(40)	7041 (27)	3964 (25)	1233 (13)	53 (21)	H(42)	8857	1620	2708	168 (61)
C(41)	8437 (29)	2944 (27)	2673 (20)	62 (22)	H(43)	9248	1472	3650	168 (61)
C(42)	8770 (29)	2161 (27)	2924 (20)	88 (27)	H(44)	9047	2714	4146	168 (61)
C(43)	8990 (29)	2078 (27)	3455 (20)	100 (29)	H(45)	8456	4102	3699	168 (61)
C(44)	8876 (29)	2778 (27)	3735 (20)	78 (25)	H(46)	8066	4250	2757	168 (61)
C(45)	8543 (29)	3561 (27)	3483 (20)	70 (23)	H(48)	8460	3130	986	168 (61)
C(46)	8324 (29)	3644 (27)	2952 (20)	38 (18)	H(49)	8553	3492	163	168 (61)
C(47)	7987 (24)	1839 (25)	737 (15)	33 (17)	H(50)	8131	2404	−549	168 (61)
C(48)	8277 (24)	2656 (25)	675 (15)	65 (23)	H(51)	7618	955	−439	168 (61)
C(49)	8328 (24)	2859 (25)	211 (15)	63 (22)	H(52)	7525	594	384	168 (61)
C(50)	8091 (24)	2246 (25)	−190 (15)	60 (21)	H(54)	9535	1020	1692	168 (61)
C(51)	7801 (24)	1430 (25)	−128 (15)	74 (24)	H(55)	10203	−423	1832	168 (61)
C(52)	7749 (24)	1226 (25)	335 (15)	45 (19)	H(56)	9433	−1798	1684	168 (61)
C(53)	8388 (31)	458 (29)	1465 (18)	56 (21)	H(57)	7994	−1730	1396	168 (61)
C(54)	9198 (31)	420 (29)	1628 (18)	72 (24)	H(58)	7326	−287	1256	168 (61)
C(55)	9575 (31)	−393 (29)	1706 (18)	113 (33)	H(60)	6415	1872	419	168 (61)
C(56)	9141 (31)	−1168 (29)	1623 (18)	68 (23)	H(61)	5047	1630	304	168 (61)
C(57)	8330 (31)	−1130 (29)	1461 (18)	103 (31)	H(62)	4781	990	1017	168 (61)
C(58)	7953 (31)	−317 (29)	1382 (18)	76 (25)	H(63)	5883	592	1843	168 (61)
C(59)	6920 (28)	1368 (26)	1195 (14)	27 (16)	H(64)	7251	833	1957	168 (61)
C(60)	6299 (28)	1592 (26)	729 (14)	57 (21)					

Table 3. *Bond lengths* (Å)

Os(2)–Os(1)	2·875 (7)	Os(3)–Os(1)	2·864 (7)	C(11)–Os(4)	1·935 (80)	O(11)–C(11)	1·126 (73)
Os(3)–Os(2)	2·895 (7)	Os(5)–Os(4)	2·900 (7)	C(12)–Os(4)	2·009 (76)	O(12)–C(12)	1·082 (71)
Os(6)–Os(4)	2·869 (6)	Os(7)–Os(4)	2·861 (7)	C(13)–Os(5)	1·925 (77)	O(13)–C(13)	1·149 (71)
Os(6)–Os(5)	2·789 (7)	Os(7)–Os(5)	2·791 (7)	C(14)–Os(5)	1·653 (76)	O(14)–C(14)	1·265 (74)
Os(7)–Os(6)	2·771 (7)	Os(8)–Os(5)	2·776 (7)	C(15)–Os(5)	2·064 (78)	O(15)–C(15)	1·046 (73)
Os(8)–Os(6)	2·794 (7)	Os(8)–Os(7)	2·777 (7)	C(16)–Os(6)	2·184 (78)	O(16)–C(16)	1·118 (72)
Os(9)–Os(5)	2·819 (7)	Os(9)–Os(6)	2·781 (7)	C(17)–Os(6)	1·917 (80)	O(17)–C(17)	1·147 (73)
Os(9)–Os(8)	2·842 (7)			C(18)–Os(6)	1·984 (80)	O(18)–C(18)	1·064 (74)
				C(19)–Os(7)	1·664 (77)	O(19)–C(19)	1·385 (77)
				C(20)–Os(7)	1·830 (79)	O(20)–C(20)	1·278 (76)
C(1)–Os(1)	1·873 (77)	O(1)–C(1)	1·158 (71)	C(21)–Os(7)	1·765 (76)	O(21)–C(21)	1·253 (74)
C(2)–Os(1)	1·827 (77)	O(2)–C(2)	1·251 (74)	C(22)–Os(8)	1·810 (80)	O(22)–C(22)	1·256 (75)
C(3)–Os(1)	1·946 (79)	O(3)–C(3)	1·165 (72)	C(23)–Os(8)	1·663 (75)	O(23)–C(23)	1·340 (74)
C(4)–Os(1)	1·860 (78)	O(4)–C(4)	1·200 (74)	C(24)–Os(8)	1·911 (79)	O(24)–C(24)	1·196 (73)
C(5)–Os(2)	1·803 (79)	O(5)–C(5)	1·186 (74)	C(25)–Os(9)	1·595 (80)	O(25)–C(25)	1·359 (78)
C(6)–Os(2)	2·127 (79)	O(6)–C(6)	1·052 (72)	C(26)–Os(9)	1·870 (77)	O(26)–C(26)	1·164 (73)
C(7)–Os(2)	1·828 (78)	O(7)–C(7)	1·201 (73)	C(27)–Os(9)	2·129 (77)	O(27)–C(27)	1·049 (87)
C(8)–Os(3)	1·707 (74)	O(8)–C(8)	1·349 (75)	O(29)–Os(2)	2·192 (42)	O(29)–C(28)	1·322 (58)
C(9)–Os(3)	1·895 (78)	O(9)–C(9)	1·152 (72)	O(28)–Os(3)	2·115 (40)	O(28)–C(28)	1·276 (56)
C(10)–Os(3)	1·752 (80)	O(10)–C(10)	1·223 (74)	C(28)–Os(4)	1·958 (55)		

Table 4. *Bond angles* (°)

Os(3)–Os(1)–Os(2)	60·6 (2)	Os(3)–Os(2)–Os(1)	59·5 (2)	C(17)–Os(6)–Os(4)	89·2 (22)	C(17)–Os(6)–Os(5)	138·3 (21)
Os(2)–Os(3)–Os(1)	59·9 (2)	Os(6)–Os(4)–Os(5)	57·8 (2)	C(17)–Os(6)–Os(7)	80·0 (22)	C(17)–Os(6)–Os(8)	112·4 (22)
Os(7)–Os(4)–Os(5)	57·9 (2)	Os(7)–Os(4)–Os(6)	57·9 (2)	C(17)–Os(6)–Os(9)	156·5 (21)	C(17)–Os(6)–C(16)	85·2 (29)
Os(6)–Os(5)–Os(4)	60·5 (2)	Os(7)–Os(5)–Os(4)	60·3 (2)	C(18)–Os(6)–Os(4)	75·4 (21)	C(18)–Os(6)–Os(5)	104·1 (22)
Os(7)–Os(5)–Os(6)	59·6 (2)	Os(8)–Os(5)–Os(6)	60·3 (2)	C(18)–Os(6)–Os(7)	136·0 (20)	C(18)–Os(6)–Os(8)	151·3 (21)
Os(8)–Os(5)–Os(7)	59·8 (2)	Os(8)–Os(5)–Os(4)	110·2 (2)	C(18)–Os(6)–Os(9)	90·3 (22)	C(18)–Os(6)–C(16)	91·5 (28)
Os(9)–Os(5)–Os(4)	112·2 (2)	Os(9)–Os(5)–Os(6)	59·5 (2)	C(18)–Os(6)–C(17)	95·5 (30)	C(18)–Os(7)–Os(4)	133·4 (24)
Os(9)–Os(5)–Os(7)	109·3 (2)	Os(9)–Os(5)–Os(8)	61·0 (2)	C(19)–Os(7)–Os(5)	151·8 (24)	C(19)–Os(7)–Os(6)	103·2 (25)
Os(5)–Os(6)–Os(4)	61·7 (2)	Os(7)–Os(6)–Os(4)	60·9 (2)	C(19)–Os(7)–Os(8)	92·5 (25)	C(20)–Os(7)–Os(4)	125·5 (23)
Os(7)–Os(6)–Os(5)	60·3 (2)	Os(8)–Os(6)–Os(5)	59·6 (2)	C(20)–Os(7)–Os(5)	99·6 (23)	C(20)–Os(7)–Os(6)	154·6 (22)
Os(8)–Os(6)–Os(7)	59·9 (2)	Os(8)–Os(6)–Os(4)	110·7 (2)	C(20)–Os(7)–Os(8)	96·9 (23)	C(20)–Os(7)–C(19)	88·2 (33)
Os(9)–Os(6)–Os(4)	114·3 (2)	Os(9)–Os(6)–Os(5)	60·8 (2)	C(21)–Os(7)–Os(4)	62·7 (24)	C(21)–Os(7)–Os(5)	118·9 (24)
Os(9)–Os(6)–Os(7)	111·0 (2)	Os(9)–Os(6)–Os(8)	61·3 (2)	C(21)–Os(7)–Os(6)	110·3 (24)	C(21)–Os(7)–Os(8)	170·5 (24)
Os(5)–Os(7)–Os(4)	61·7 (2)	Os(6)–Os(7)–Os(4)	61·2 (2)	C(21)–Os(7)–C(19)	87·5 (33)	C(21)–Os(7)–C(20)	92·6 (33)
Os(6)–Os(7)–Os(5)	60·2 (2)	Os(8)–Os(7)–Os(4)	111·4 (2)	C(22)–Os(8)–Os(5)	96·8 (23)	C(22)–Os(8)–Os(6)	146·0 (22)
Os(8)–Os(7)–Os(5)	59·8 (2)	Os(8)–Os(7)–Os(6)	60·5 (2)	C(22)–Os(8)–Os(7)	87·8 (23)	C(22)–Os(8)–Os(9)	133·7 (22)
Os(6)–Os(8)–Os(5)	60·1 (2)	Os(7)–Os(8)–Os(5)	60·3 (2)	C(23)–Os(8)–Os(5)	113·1 (25)	C(23)–Os(8)–Os(6)	128·9 (25)
Os(7)–Os(8)–Os(6)	59·7 (2)	Os(9)–Os(8)–Os(5)	60·2 (2)	C(23)–Os(8)–Os(7)	166·8 (24)	C(23)–Os(8)–Os(9)	73·7 (25)
Os(9)–Os(8)–Os(6)	59·1 (2)	Os(9)–Os(8)–Os(7)	109·0 (2)	C(23)–Os(8)–C(22)	81·6 (33)	C(24)–Os(8)–Os(5)	154·1 (21)
Os(6)–Os(9)–Os(5)	59·7 (2)	Os(8)–Os(9)–Os(5)	58·7 (2)	C(24)–Os(8)–Os(6)	98·2 (22)	C(24)–Os(8)–Os(7)	97·5 (22)
Os(8)–Os(9)–Os(6)	59·6 (2)			C(24)–Os(8)–Os(9)	123·2 (22)	C(24)–Os(8)–C(22)	95·3 (31)
				C(24)–Os(8)–C(23)	91·3 (32)	C(25)–Os(9)–Os(5)	100·4 (26)
				C(25)–Os(9)–Os(6)	156·3 (25)	C(25)–Os(9)–Os(8)	100·0 (26)
C(1)–Os(1)–Os(2)	88·2 (23)	C(1)–Os(1)–Os(3)	86·5 (22)	C(26)–Os(9)–Os(5)	103·8 (22)	C(26)–Os(9)–Os(6)	95·8 (23)
C(2)–Os(1)–Os(2)	86·1 (23)	C(2)–Os(1)–Os(3)	88·5 (23)	C(26)–Os(9)–Os(8)	154·2 (21)	C(26)–Os(9)–C(25)	102·0 (35)
C(2)–Os(1)–C(1)	173·8 (31)	C(3)–Os(1)–Os(2)	102·8 (21)	C(27)–Os(9)–Os(5)	166·1 (19)	C(27)–Os(9)–Os(6)	109·1 (21)
C(3)–Os(1)–Os(3)	163·3 (20)	C(3)–Os(1)–C(1)	95·3 (30)	C(27)–Os(9)–Os(8)	109·3 (20)	C(27)–Os(9)–C(25)	88·2 (32)
C(3)–Os(1)–C(2)	88·5 (31)	C(4)–Os(1)–Os(2)	161·4 (22)	C(27)–Os(9)–C(26)	84·7 (30)		
C(4)–Os(1)–Os(3)	101·1 (23)	C(4)–Os(1)–C(1)	94·3 (31)				
C(4)–Os(1)–C(2)	90·2 (31)	C(4)–Os(1)–C(3)	95·3 (31)				
C(5)–Os(2)–Os(1)	87·4 (24)	C(5)–Os(2)–Os(3)	96·9 (23)	O(1)–C(1)–Os(1)	167·6 (65)	O(2)–C(2)–Os(1)	173·2 (60)
C(6)–Os(2)–Os(1)	86·4 (20)	C(6)–Os(2)–Os(3)	145·0 (19)	O(3)–C(3)–Os(1)	167·3 (64)	O(4)–C(4)–Os(1)	173·3 (64)
C(6)–Os(2)–C(5)	88·5 (30)	C(7)–Os(2)–Os(1)	176·2 (22)	O(5)–C(5)–Os(2)	172·7 (67)	O(6)–C(6)–Os(2)	170·8 (70)
C(7)–Os(2)–Os(3)	123·0 (23)	C(7)–Os(2)–C(5)	94·9 (33)	O(7) C(7) Os(2)	172·2 (63)	O(8) C(8)–Os(3)	153·0 (57)
C(7)–Os(2)–C(6)	90·7 (30)	C(8)–Os(3)–Os(1)	87·9 (24)	O(9)–C(9)–Os(3)	173·8 (66)	O(10)–C(10)–Os(3)	172·3 (63)
C(8)–Os(3)–Os(2)	106·6 (24)	C(9)–Os(3)–Os(1)	86·4 (22)	O(11)–C(11)–Os(4)	176·6 (68)	O(12)–C(12)–Os(4)	161·7 (68)
C(9)–Os(3)–Os(2)	143·7 (21)	C(9)–Os(3)–C(8)	83·6 (32)	O(13)–C(13)–Os(5)	170·5 (64)	O(14)–C(14)–Os(5)	171·3 (60)
C(10)–Os(3)–Os(1)	173·7 (22)	C(10)–Os(3)–Os(2)	115·2 (24)	O(15)–C(15)–Os(5)	172·2 (73)	O(16)–C(16)–Os(6)	154·8 (64)
C(10)–Os(3)–C(8)	97·5 (33)	C(10)–Os(3)–C(9)	97·3 (32)	O(17)–C(17)–Os(6)	168·6 (54)	O(18)–C(18)–Os(6)	178·8 (71)
C(11)–Os(4)–Os(5)	104·2 (22)	C(11)–Os(4)–Os(6)	108·1 (22)	O(19)–C(19)–Os(7)	168·7 (59)	O(20)–C(20)–Os(7)	165·3 (61)
C(11)–Os(4)–Os(7)	160·8 (21)	C(12)–Os(4)–Os(5)	104·5 (21)	O(21)–C(21)–Os(4)	117·4 (51)	O(22)–C(22)–Os(8)	174·0 (61)
C(12)–Os(4)–Os(6)	155·3 (20)	C(12)–Os(4)–Os(7)	98·9 (21)	O(23)–C(23)–Os(8)	170·8 (60)	O(24)–C(24)–Os(8)	168·2 (62)
C(12)–Os(4)–C(11)	92·3 (30)	C(13)–Os(5)–Os(4)	75·6 (22)	O(25)–C(25)–Os(9)	159·8 (60)	O(26)–C(26)–Os(9)	175·2 (66)
C(13)–Os(5)–Os(6)	130·3 (21)	C(13)–Os(5)–Os(7)	79·7 (22)	O(27)–C(27)–Os(9)	164·0 (79)		
C(13)–Os(5)–Os(8)	123·1 (21)	C(13)–Os(5)–Os(9)	170·1 (21)				
C(14)–Os(5)–Os(4)	167·1 (23)	C(14)–Os(5)–Os(6)	132·1 (23)				
C(14)–Os(5)–Os(7)	120·9 (25)	C(14)–Os(5)–Os(8)	78·9 (25)	O(28)–C(28)–Os(4)	129·0 (43)	O(29)–C(28)–Os(4)	117·7 (40)
C(14)–Os(5)–Os(9)	80·1 (25)	C(14)–Os(5)–C(13)	91·8 (32)	O(29)–C(28)–O(28)	112·4 (48)	C(28)–O(28)–Os(3)	133·4 (35)
C(15)–Os(5)–Os(4)	79·1 (21)	C(15)–Os(5)–Os(6)	101·8 (22)	C(28)–O(29)–Os(2)	132·7 (34)	O(29)–Os(2)–Os(1)	93·7 (11)
C(15)–Os(5)–Os(7)	139·4 (20)	C(15)–Os(5)–Os(8)	145·8 (20)	O(29)–Os(2)–Os(3)	78·6 (11)	O(29)–Os(2)–C(5)	173·9 (25)
C(15)–Os(5)–Os(9)	84·8 (21)	C(15)–Os(5)–C(13)	90·9 (30)	O(29)–Os(2)–C(6)	97·5 (23)	O(29)–Os(2)–C(7)	84·3 (26)
C(15)–Os(5)–C(14)	98·6 (32)	C(16)–Os(6)–Os(4)	165·2 (18)	O(28)–Os(3)–Os(1)	92·2 (11)	O(28)–Os(3)–Os(2)	81·7 (11)
C(16)–Os(6)–Os(5)	129·8 (20)	C(16)–Os(6)–Os(7)	131·0 (19)	O(28)–Os(3)–C(8)	170·3 (25)	O(28)–Os(3)–C(9)	86·7 (25)
C(16)–Os(6)–Os(8)	84·2 (19)	C(16)–Os(6)–Os(9)	71·9 (20)	O(28)–Os(3)–C(10)	83·0 (26)	C(28)–Os(4)–Os(5)	165·6 (16)
				C(28)–Os(4)–Os(6)	108·8 (17)	C(28)–Os(4)–Os(7)	111·9 (17)
				C(28)–Os(4)–C(11)	84·2 (27)	C(28)–Os(4)–C(12)	86·5 (27)

Table 5. *Selected bond lengths* (Å) *and angles* (°) *in the* $[(Ph_3P)_2N]^+$ *cation*

N–P(1)	1·573 (57)	N–P(2)	1·598 (56)
C(29)–P(1)	1·806 (48)	C(47)–P(2)	1·798 (48)
C(35)–P(1)	1·802 (49)	C(53)–P(2)	1·762 (58)
C(41)–P(1)	1·776 (55)	C(59)–P(2)	1·769 (50)
P(2)–N–P(1)	136·6 (37)	C(47)–P(2)–N	112·5 (24)
C(29)–P(1)–N	109·8 (25)	C(53)–P(2)–N	109·6 (27)
C(35)–P(1)–N	117·7 (27)	C(59)–P(2)–N	111·7 (26)
C(41)–P(1)–N	108·5 (25)	C(53)–P(2)–C(47)	104·9 (22)
C(35)–P(1)–C(29)	106·3 (20)	C(59)–P(2)–C(47)	108·4 (20)
C(41)–P(1)–C(29)	105·6 (23)	C(59)–P(2)–C(53)	109·6 (22)
C(41)–P(1)–C(35)	108·3 (21)		

groups [Os(7) and Os(9) axial, Os(5), Os(6) and Os(8) equatorial] with one triangular face capped by Os(4). The Os–Os distances within this cluster are similar to those found in $Os_6(CO)_{18}$ (Mason, Thomas & Mingos, 1973), except for the bonds to Os(4) which are about 0·05 Å longer. The CO_2 bridge attached to Os(4) may be regarded as either a carbene or a carboxylato substituent; spectroscopic evidence indicates that no H atom is attached to the CO_2 unit (Eady *et al.*, 1976). From NMR evidence and the positions occupied by the carbonyl groups, the most likely position for the hydride is bridging the edge Os(2)–Os(3) of the Os_3

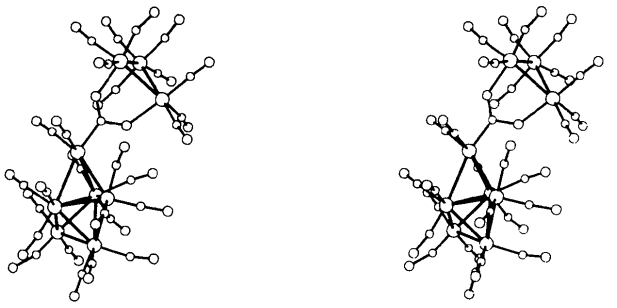

Fig. 2. Stereoscopic view of the $[HOs_3(CO)_{10}.O_2C.Os_6(CO)_{17}]^-$ anion.

cluster. The ring Os(2)–O(29)–C(28)–O(28)–Os(3) is virtually symmetrical and planar; the stability of the complex may be enhanced by some electron delocalization in the ring. The cation dimensions are similar to those reported by Handy, Ruff & Dahl (1970).

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References

EADY, C. R., GUY, J. J., JOHNSON, B. F. G., LEWIS, J., MALATESTA, M. C. & SHELDRICK, G. M. (1976). *J. Chem. Soc. Chem. Commun.* pp. 602–604.

HANDY, L. B., RUFF, J. K. & DAHL, L. F. (1970). *J. Am. Chem. Soc.* **92**, 7312–7326.

MASON, R., THOMAS, K. M. & MINGOS, D. M. P. (1973). *J. Am. Chem. Soc.* **95**, 3802–3804.

Acta Cryst. (1978). B**34**, 1722–1725

Bis(triphenylphosphine)iminium μ-Hydrido-pentadecacarbonylpentaosmate, $[(Ph_3P)_2N]^+[HOs_5(CO)_{15}]^-$

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Abstract. $C_{51}H_{31}NO_{15}P_2Os_5$, monoclinic, $C2/c$, a = 21·98 (1), b = 15·76 (1), c = 31·01 (2) Å, β = 99·3 (1)°, U = 10 596 Å^3, Z = 8, D_x = 2·395 g cm^{-3}, μ(Mo $K\alpha$) = 116·2 cm^{-1}. The structure was refined to an R of 0·060 for 6001 unique diffractometer data. The anion consists of a trigonal-bipyramidal cluster of five Os atoms, with three carbonyls attached to each; the hydride probably bridges an equatorial Os–Os bond.

Introduction. The $[HOs_5(CO)_{15}]^-$ anion can be prepared by the action of excess isopropylamine on $Os_6(CO)_{18}$, and isolated as the $[(Ph_3P)_2N]^+$ salt (Eady, Guy, Johnson, Lewis, Malatesta & Sheldrick, 1976).

Red-black crystals were grown by diffusion of cyclohexane into a dilute solution of the compound in dichloromethane. 9211 reflexions in the range $1·0 < 2\theta < 55·0°$ were measured with a Syntex $P2_1$ four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation and a crystal 0·25 × 0·16 × 0·06 mm. Numerical absorption corrections were applied, and equivalent reflexions averaged to give 6001 unique data with $F_o > 4\sigma(F)$ based on counting statistics. Os atoms were located by multisolution $\sum_2$ sign expansion, and the remaining non-hydrogen atoms from difference syntheses. The structure was refined by blocked-cascade least squares with rigid idealized phenyl groups [C–C 1·395 Å; U(H) fixed at 0·10 Å^2; C–H 1·08 Å and all angles at C 120°], anisotropic temperature factors for Os and the remaining atoms isotropic, and the weighting scheme $w = [\sigma^2(F) + 0·0012F^2]^{-1}$ which gave a virtually flat analysis of variance; complex neutral-atom scattering factors were employed. The final $R' = \sum w^{1/2}\Delta/\sum w^{1/2}F_o$ was 0·059 and R = 0·060. Atomic coordinates and isotropic temperature factors are given in Table 1, bond lengths and angles for the anion in Tables 2 and 3, and selected dimensions for the cation in Table 4. Fig. 1 shows the atom labelling.*

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33349 (38 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.