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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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, $\beta = 99.3$ (1)°, $U = 10\,596$ Å^3, $Z = 8$, $D_x = 2.395$ g cm^{-3}, $\mu(Mo\ K\alpha) = 116.2$ 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 $0.25 \times 0.16 \times 0.06$ 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.*

Table 1. *Atom coordinates* ($\times 10^4$) *and isotropic temperature factors* ($\text{Å}^2 \times 10^3$)

	x	y	z	U		x	y	z	U
Os(1)	752 (1)	1559 (1)	4895 (1)		C(30)	3202 (7)	2749 (7)	3530 (5)	85 (8)
Os(2)	792 (1)	3127 (1)	4455 (1)		C(31)	3401 (7)	3529 (7)	3391 (5)	59 (6)
Os(3)	−283 (1)	2112 (1)	4275 (1)		C(32)	3409 (7)	3670 (7)	2948 (5)	85 (8)
Os(4)	67 (1)	3014 (1)	5082 (1)		C(33)	3218 (7)	3032 (7)	2644 (5)	74 (7)
Os(5)	834 (1)	1661 (1)	3991 (1)		C(34)	3768 (6)	2148 (7)	1601 (5)	42 (5)
C(1)	887 (8)	420 (12)	4775 (7)	48 (5)	C(35)	4108 (6)	1424 (7)	1537 (5)	56 (6)
O(1)	978 (7)	−310 (11)	4736 (6)	82 (5)	C(36)	4751 (6)	1462 (7)	1597 (5)	69 (7)
C(2)	547 (9)	1320 (13)	5455 (7)	52 (5)	C(37)	5053 (6)	2224 (7)	1719 (5)	69 (7)
O(2)	437 (7)	1129 (10)	5796 (6)	71 (4)	C(38)	4713 (6)	2948 (7)	1782 (5)	70 (7)
C(3)	1575 (9)	1839 (12)	5112 (7)	46 (5)	C(39)	4071 (6)	2910 (7)	1723 (5)	70 (7)
O(3)	2089 (7)	1936 (10)	5248 (5)	65 (4)	C(40)	2641 (6)	3120 (8)	1322 (4)	39 (4)
C(4)	1353 (10)	3302 (15)	4056 (8)	63 (6)	C(41)	2124 (6)	3438 (8)	1475 (4)	48 (5)
O(4)	1707 (8)	3475 (11)	3833 (6)	81 (5)	C(42)	1862 (6)	4204 (8)	1314 (4)	61 (6)
C(5)	418 (10)	4140 (16)	4260 (8)	66 (6)	C(43)	2117 (6)	4652 (8)	999 (4)	58 (6)
O(5)	163 (9)	4751 (13)	4115 (7)	102 (6)	C(44)	2634 (6)	4334 (8)	845 (4)	61 (6)
C(6)	1357 (9)	3635 (13)	4905 (7)	52 (5)	C(45)	2896 (6)	3568 (8)	1007 (4)	50 (5)
O(6)	1703 (7)	3938 (10)	5162 (5)	64 (4)	C(46)	2722 (6)	1371 (9)	1062 (4)	45 (5)
C(7)	−473 (10)	2862 (15)	3805 (8)	66 (6)	C(47)	2291 (6)	734 (9)	1096 (4)	49 (5)
O(7)	−587 (8)	3271 (11)	3499 (6)	82 (5)	C(48)	2137 (6)	152 (9)	757 (4)	78 (7)
C(8)	−578 (10)	1151 (15)	3925 (8)	62 (6)	C(49)	2413 (6)	207 (9)	384 (4)	67 (6)
O(8)	−786 (8)	593 (12)	3739 (6)	82 (5)	C(50)	2843 (6)	843 (9)	349 (4)	79 (7)
C(9)	−1030 (10)	2294 (14)	4494 (7)	57 (6)	C(51)	2998 (6)	1425 (9)	688 (4)	73 (7)
O(9)	−1511 (8)	2397 (11)	4601 (6)	80 (5)	H(17)	2696 (6)	−156 (11)	1961 (5)	100
C(10)	554 (9)	3471 (14)	5588 (7)	53 (5)	H(18)	3406 (6)	−1386 (11)	2020 (5)	100
O(10)	849 (7)	3747 (10)	5871 (6)	70 (4)	H(19)	4385 (6)	−1347 (11)	2541 (5)	100
C(11)	−478 (10)	2541 (14)	5422 (7)	56 (6)	H(20)	4656 (6)	−78 (11)	3003 (5)	100
O(11)	−801 (6)	2213 (9)	5620 (5)	58 (4)	H(21)	3947 (6)	1152 (11)	2944 (5)	100
C(12)	−440 (9)	3970 (14)	4935 (7)	55 (6)	H(23)	1486 (6)	1733 (8)	1984 (4)	100
O(12)	−729 (7)	4563 (11)	4864 (6)	76 (5)	H(24)	495 (6)	1206 (8)	2162 (4)	100
C(13)	1694 (9)	1507 (13)	4009 (7)	53 (5)	H(25)	488 (6)	219 (8)	2782 (4)	100
O(13)	2215 (8)	1458 (10)	4024 (6)	78 (5)	H(26)	1473 (6)	−241 (8)	3224 (4)	100
C(14)	665 (10)	2124 (14)	3407 (7)	58 (6)	H(27)	2464 (6)	286 (8)	3046 (4)	100
O(14)	562 (8)	2387 (11)	3068 (6)	84 (5)	H(29)	2858 (7)	1509 (7)	3335 (5)	100
C(15)	669 (9)	545 (14)	3825 (8)	57 (6)	H(30)	3195 (7)	2642 (7)	3873 (5)	100
O(15)	536 (7)	−137 (11)	3699 (6)	74 (5)	H(31)	3548 (7)	4024 (7)	3625 (5)	100
P(1)	2749 (2)	1457 (3)	2383 (2)	36 (1)	H(32)	3563 (7)	4272 (7)	2838 (5)	100
P(2)	2936 (2)	2095 (3)	1505 (2)	35 (1)	H(33)	3225 (7)	3139 (7)	2300 (5)	100
N(1)	2625 (7)	1822 (10)	1911 (5)	44 (4)	H(35)	3874 (6)	834 (7)	1443 (5)	100
C(16)	3277 (6)	578 (11)	2447 (5)	35 (4)	H(36)	5014 (6)	900 (7)	1549 (5)	100
C(17)	3124 (6)	−139 (11)	2188 (5)	95 (9)	H(37)	5551 (6)	2252 (7)	1766 (5)	100
C(18)	3525 (6)	−831 (11)	2221 (5)	87 (8)	H(38)	4948 (6)	3538 (7)	1877 (5)	100
C(19)	4078 (6)	−807 (11)	2513 (5)	78 (7)	H(39)	3808 (6)	3472 (7)	1770 (5)	100
C(20)	4231 (6)	−91 (11)	2772 (5)	126 (12)	H(41)	1926 (6)	3091 (8)	1719 (4)	100
C(21)	3831 (6)	602 (11)	2739 (5)	91 (9)	H(42)	1462 (6)	4451 (8)	1434 (4)	100
C(22)	2038 (6)	1044 (8)	2504 (4)	38 (4)	H(43)	1914 (6)	5246 (8)	875 (4)	100
C(23)	1483 (6)	1303 (8)	2255 (4)	61 (6)	H(44)	2831 (6)	4681 (8)	601 (4)	100
C(24)	925 (6)	1004 (8)	2356 (4)	73 (7)	H(45)	3296 (6)	3321 (8)	886 (4)	100
C(25)	922 (6)	447 (8)	2705 (4)	64 (6)	H(47)	2079 (6)	691 (9)	1387 (4)	100
C(26)	1478 (6)	189 (8)	2954 (4)	50 (5)	H(48)	1803 (6)	−342 (9)	785 (4)	100
C(27)	2036 (6)	488 (8)	2853 (4)	44 (5)	H(49)	2289 (6)	−242 (9)	120 (4)	100
C(28)	3019 (7)	2253 (7)	2783 (5)	33 (4)	H(50)	3050 (6)	889 (9)	57 (4)	100
C(29)	3011 (7)	2112 (7)	3226 (5)	71 (7)	H(51)	3326 (6)	1922 (9)	658 (4)	100

Table 2. *Bond lengths* (Å)

Os(1)—Os(2)	2·831 (4)	Os(2)—C(6)	1·888 (22)	Os(1)—Os(3)	2·867 (4)	C(6)—O(6)	1·115 (26)
Os(2)—Os(3)	2·833 (4)	Os(3)—C(7)	1·870 (27)	Os(1)—Os(5)	2·843 (4)	C(7)—O(7)	1·141 (32)
Os(2)—Os(5)	2·732 (4)	Os(3)—C(8)	1·913 (25)	Os(3)—Os(5)	2·829 (4)	C(8)—O(8)	1·112 (30)
Os(1)—Os(4)	2·852 (4)	Os(3)—C(9)	1·898 (25)	Os(2)—Os(4)	2·710 (4)	C(9)—O(9)	1·168 (30)
Os(3)—Os(4)	2·872 (4)	Os(4)—C(10)	1·893 (23)			C(10)—O(10)	1·094 (27)
Os(1)—C(1)	1·867 (22)	Os(4)—C(11)	1·876 (25)	C(1)—O(1)	1·177 (28)	C(11)—O(11)	1·135 (29)
Os(1)—C(2)	1·900 (25)	Os(4)—C(12)	1·885 (23)	C(2)—O(2)	1·161 (30)	C(12)—O(12)	1·132 (28)
Os(1)—C(3)	1·879 (21)	Os(5)—C(13)	1·898 (23)	C(3)—O(3)	1·149 (25)	C(13)—O(13)	1·140 (28)
Os(2)—C(4)	1·903 (28)	Os(5)—C(14)	1·933 (24)	C(4)—O(4)	1·153 (33)	C(14)—O(14)	1·119 (29)
Os(2)—C(5)	1·852 (26)	Os(5)—C(15)	1·851 (24)	C(5)—O(5)	1·166 (32)	C(15)—O(15)	1·166 (29)

Table 3. Bond angles (°)

Os(1)—Os(2)—Os(3)	60·8 (1)	Os(1)—Os(3)—Os(2)	59·5 (1)
Os(2)—Os(1)—Os(3)	59·6 (1)	Os(1)—Os(4)—Os(2)	61·1 (1)
Os(1)—Os(4)—Os(3)	60·1 (1)	Os(2)—Os(4)—Os(3)	60·9 (1)
Os(2)—Os(1)—Os(4)	57·0 (1)	Os(1)—Os(2)—Os(4)	61·9 (1)
Os(4)—Os(2)—Os(3)	62·4 (1)	Os(2)—Os(3)—Os(4)	56·7 (1)
Os(4)—Os(1)—Os(3)	60·3 (1)	Os(1)—Os(3)—Os(4)	59·6 (1)
Os(2)—Os(1)—Os(5)	57·6 (1)	Os(1)—Os(2)—Os(5)	61·4 (1)
Os(5)—Os(1)—Os(3)	59·4 (1)	Os(1)—Os(3)—Os(5)	59·9 (1)
Os(5)—Os(2)—Os(3)	61·1 (1)	Os(2)—Os(3)—Os(5)	57·7 (1)
Os(1)—Os(5)—Os(2)	61·0 (1)	Os(1)—Os(5)—Os(3)	60·7 (1)
Os(2)—Os(5)—Os(3)	61·2 (1)	Os(5)—Os(1)—Os(4)	105·9 (1)
Os(5)—Os(2)—Os(4)	113·3 (1)	Os(5)—Os(3)—Os(4)	105·7 (1)
Os(2)—Os(1)—C(1)	136·4 (7)	Os(3)—Os(1)—C(1)	107·0 (7)
Os(4)—Os(1)—C(1)	156·9 (6)	Os(5)—Os(1)—C(1)	79·6 (7)
Os(2)—Os(1)—C(2)	130·1 (6)	Os(3)—Os(1)—C(2)	113·2 (7)
Os(4)—Os(1)—C(2)	76·3 (7)	Os(5)—Os(1)—C(2)	167·0 (6)
Os(2)—Os(1)—C(3)	82·2 (7)	Os(3)—Os(1)—C(3)	141·5 (6)
Os(4)—Os(1)—C(3)	104·4 (7)	Os(5)—Os(1)—C(3)	97·4 (8)
C(1)—Os(1)—C(2)	93·5 (10)	C(1)—Os(1)—C(3)	97·0 (9)
C(2)—Os(1)—C(3)	94·3 (10)	Os(1)—Os(2)—C(4)	121·0 (8)
Os(3)—Os(2)—C(4)	124·0 (8)	Os(4)—Os(2)—C(4)	173·5 (7)
Os(5)—Os(2)—C(4)	72·5 (8)	Os(1)—Os(2)—C(5)	149·6 (8)
Os(3)—Os(2)—C(5)	96·3 (8)	Os(4)—Os(2)—C(5)	90·5 (9)
Os(5)—Os(2)—C(5)	127·4 (9)	Os(1)—Os(2)—C(6)	94·7 (8)
Os(3)—Os(2)—C(6)	144·4 (7)	Os(4)—Os(2)—C(6)	83·7 (8)
Os(5)—Os(2)—C(6)	132·4 (6)	C(4)—Os(2)—C(5)	87·9 (12)
C(4)—Os(2)—C(6)	90·1 (11)	C(5)—Os(2)—C(6)	94·6 (11)
Os(1)—Os(3)—C(7)	140·2 (7)	Os(2)—Os(3)—C(7)	82·6 (8)
Os(4)—Os(3)—C(7)	111·2 (8)	Os(5)—Os(3)—C(7)	90·7 (9)
Os(1)—Os(3)—C(8)	107·4 (7)	Os(2)—Os(3)—C(8)	138·9 (7)
Os(4)—Os(3)—C(8)	154·7 (7)	Os(5)—Os(3)—C(8)	81·7 (8)
Os(1)—Os(3)—C(9)	116·3 (7)	Os(2)—Os(3)—C(9)	125·9 (7)
Os(4)—Os(3)—C(9)	74·8 (7)	Os(5)—Os(3)—C(9)	173·6 (6)
C(7)—Os(3)—C(8)	92·6 (11)	C(7)—Os(3)—C(9)	95·0 (11)
C(8)—Os(3)—C(9)	95·1 (10)	Os(1)—Os(4)—C(10)	103·2 (7)
Os(2)—Os(4)—C(10)	104·5 (8)	Os(3)—Os(4)—C(10)	161·1 (7)
Os(1)—Os(4)—C(11)	101·6 (8)	Os(2)—Os(4)—C(11)	158·9 (7)
Os(3)—Os(4)—C(11)	100·8 (8)	Os(1)—Os(4)—C(12)	154·7 (7)
Os(2)—Os(4)—C(12)	99·7 (8)	Os(3)—Os(4)—C(12)	96·9 (8)
C(10)—Os(4)—C(11)	90·7 (11)	C(10)—Os(4)—C(12)	97·4 (10)
C(11)—Os(4)—C(12)	92·6 (11)	Os(1)—Os(5)—C(13)	100·7 (8)
Os(2)—Os(5)—C(13)	102·2 (7)	Os(3)—Os(5)—C(13)	159·0 (6)
Os(1)—Os(5)—C(14)	155·8 (7)	Os(2)—Os(5)—C(14)	99·0 (7)
Os(3)—Os(5)—C(14)	98·7 (8)	Os(1)—Os(5)—C(15)	100·4 (8)
Os(2)—Os(5)—C(15)	158·1 (7)	Os(3)—Os(5)—C(15)	100·6 (8)
C(13)—Os(5)—C(14)	96·6 (10)	C(13)—Os(5)—C(15)	92·1 (10)
C(14)—Os(5)—C(15)	95·7 (10)		
Os(1)—C(1)—O(1)	174·3 (19)	Os(1)—C(2)—O(2)	176·1 (18)
Os(1)—C(3)—O(3)	174·1 (18)	Os(2)—C(4)—O(4)	173·9 (21)
Os(2)—C(5)—O(5)	175·8 (19)	Os(2)—C(6)—O(6)	177·9 (21)
Os(3)—C(7)—O(7)	175·1 (22)	Os(3)—C(8)—O(8)	174·6 (22)
Os(3)—C(9)—O(9)	175·4 (18)	Os(4)—C(10)—O(10)	177·5 (23)
Os(4)—C(11)—O(11)	176·3 (19)	Os(4)—C(12)—O(12)	176·7 (21)
Os(5)—C(13)—O(13)	176·5 (20)	Os(5)—C(14)—O(14)	179·3 (13)
Os(5)—C(15)—O(15)	175·5 (15)		
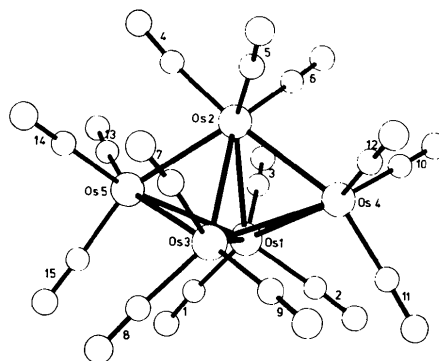
Fig. 1. The |HOs₅(CO)₁₅]⁻ anion, showing the labelling scheme.

Discussion. The five Os atoms form a trigonal bipyramid, as in Os₅(CO)₁₆ (Reichert & Sheldrick, 1977); replacement of a terminal carbonyl attached to an equatorial Os atom by a (presumably bridging) hydride has left the cluster geometry unchanged. Although the shortest Os···C non-bonded contact [Os(5)···C(4) 2·82 Å] is longer than the shortest contact in Os₅(CO)₁₆ (2·68 Å), it is associated with the smallest Os—C—O angle (173·9°) which bends away from Os(5), so it might be considered to be a weak incipient carbonyl bridge, serving to equalize the oxidation states (or connectivities) of Os(2) and Os(5). The two shortest Os—Os bonds [Os(2)—Os(4) 2·710, Os(2)—Os(5) 2·732 Å] link Os atoms of different oxidation states in contrast to Os₆(CO)₁₈ (Mason, Thomas & Mingos, 1973) and most other Os carbonyl derivatives. A three-dimensional view of the molecule (Fig. 2) shows that the carbonyl groups are approximately staggered, except about Os(1)—Os(3) where they are eclipsed, and that there is also a hole in the close-packed carbonyl foliage consistent with the presence of a hydride bridging this bond. This assignment is consistent with the ¹H and ¹³C NMR data (Eady et al., 1976). Although single hydride bridges are normally found across the longest Os—Os bond in related compounds, Os(3)—Os(4) is slightly longer [2·872 (4) compared with 2·867 (4) Å]. A
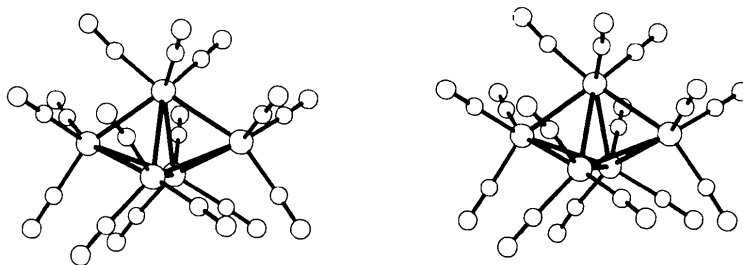

Fig. 2. Stereoscopic view of the [HOs₅(CO)₁₅]⁻ anion.

Table 4. *Selected bond lengths* (Å) *and angles* (°) *in the* $[(Ph_3P)_2N]^+$ *cation*

P(1)–N(1)	1·554 (18)	P(2)–N(1)	1·586 (19)
P(1)–C(16)	1·797 (18)	P(2)–C(34)	1·808 (15)
P(1)–C(22)	1·788 (16)	P(2)–C(40)	1·798 (15)
P(1)–C(28)	1·797 (16)	P(2)–C(46)	1·790 (15)
P(1)–N(1)–P(2)	144·5 (10)	N(1)–P(2)–C(34)	115·9 (8)
N(1)–P(1)–C(16)	113·8 (9)	N(1)–P(2)–C(40)	108·4 (8)
N(1)–P(1)–C(22)	107·7 (8)	N(1)–P(2)–C(46)	110·1 (8)
N(1)–P(1)–C(28)	112·4 (8)	C(34)–P(2)–C(40)	108·3 (7)
C(16)–P(1)–C(22)	105·4 (8)	C(34)–P(2)–C(46)	106·8 (8)
C(16)–P(1)–C(28)	109·1 (8)	C(40)–P(2)–C(46)	107·1 (7)
C(22)–P(1)–C(28)	108·1 (8)		

nominal 18-electron valence-shell configuration can be achieved for each Os atom by putting -1 charges on Os(4) and Os(5), and considering the Os–H–Os unit

to be linked by a two-electron three-centre bond, with a $+1$ charge on the H atom.

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Di-μ-hydrido-hexadecacarbonylpentaosmium

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Abstract. $C_{16}H_2O_{16}Os_5$, triclinic, $P\bar{1}$, $a = 8·99$ (1), $b = 10·60$ (1), $c = 13·32$ (1) Å, $\alpha = 100·2$ (1), $\beta = 90·2$ (1), $\gamma = 106·7$ (1)°, $M_r = 1401·2$, $Z = 2$, $D_x = 3·90$ g cm^{-3}, $U = 1194·1$ Å^3, μ(Mo $K\alpha$) = 256·2 cm^{-1}. The structure was refined to an R of 0·078 for 3408 unique diffractometer data, and revealed a novel Os$_5$ cluster geometry based on an edge-bridged tetrahedron.

Introduction. The title compound was first observed as a minor product of the pyrolysis of $Os_3(CO)_{12}$ in the presence of water by Eady, Johnson & Lewis (1973, 1977). Wade's (1975) theory would predict a square-based pyramidal arrangement of the five Os atoms, in contrast to the structure reported here.

Brown laminar crystals [principal faces (001)] were grown by slow diffusion of pentane into a dichloromethane solution. 5513 unique data were collected with a Syntex $P2_1$ four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation, and a crystal 0·23 × 0·23 × 0·01 mm. Numerical absorption corrections were applied. The Os atoms were located by multisolution $\sum_2$ sign expansion, and the C and O atoms from difference

syntheses. The structure was refined by blocked-cascade least squares to $R' = \sum w^{1/2}\Delta/\sum w^{1/2}F_o = 0·078$ and $R = 0·078$ for the 3408 reflexions with $F_o > 5\sigma(F)$ based on counting statistics. Complex neutral-atom scattering factors were employed, with aniso-
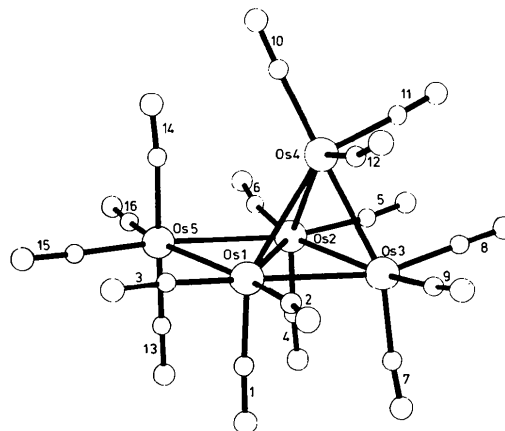

Fig. 1. $H_2Os_5(CO)_{16}$, showing the numbering scheme.