Table 4. *Selected bond lengths* (Å) *and angles* (°) *in the* [(Ph₃P)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, $U = 1194·1$ Å³, $\mu(\text{Mo } K\alpha) = 256·2$ 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-
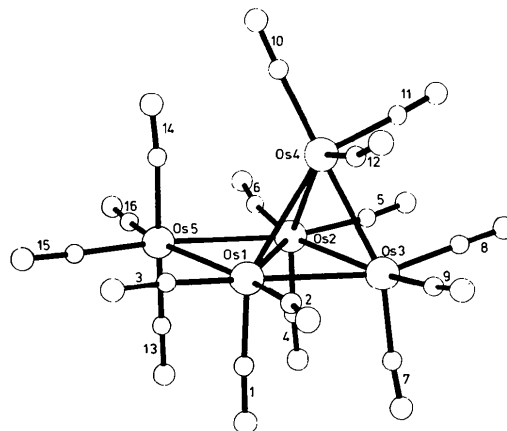

Fig. 1. $H_2Os_5(CO)_{16}$, showing the numbering scheme.

Table 1. Atom coordinates (×10⁴) and isotropic temperature factors (Å² ×10³)

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

	x	y	z	U
Os(1)	1008 (1)	6601 (1)	2525 (1)	
Os(2)	4204 (1)	7375 (1)	3035 (1)	
Os(3)	2847 (2)	9244 (1)	2408 (1)	
Os(4)	3191 (2)	7190 (1)	887 (1)	
Os(5)	2354 (1)	4694 (1)	3190 (1)	
C(1)	385 (42)	7167 (37)	3805 (30)	38 (8)
O(1)	49 (30)	7625 (27)	4665 (22)	46 (6)
C(2)	−327 (44)	7444 (39)	1965 (31)	42 (9)
O(2)	−1305 (34)	7731 (30)	1613 (24)	55 (7)
C(3)	−609 (39)	5044 (35)	2314 (28)	34 (7)
O(3)	−1722 (33)	4095 (30)	2219 (23)	54 (7)
C(4)	3994 (39)	8081 (35)	4405 (29)	33 (7)
O(4)	3893 (31)	8567 (28)	5218 (23)	50 (7)
C(5)	5894 (40)	8906 (35)	2928 (28)	34 (8)
O(5)	7078 (35)	9701 (32)	2905 (25)	59 (8)
C(6)	5654 (43)	6517 (39)	3384 (31)	41 (8)
O(6)	6702 (40)	6110 (35)	3518 (28)	69 (9)
C(7)	2450 (42)	10120 (38)	3726 (31)	39 (8)
O(7)	2253 (36)	10685 (33)	4434 (26)	61 (8)
C(8)	4474 (43)	10653 (38)	2127 (31)	40 (8)
O(8)	5503 (39)	11508 (34)	1998 (27)	68 (9)
C(9)	1683 (55)	10046 (49)	1728 (40)	61 (12)
O(9)	860 (41)	10389 (36)	1212 (29)	73 (10)
C(10)	3433 (37)	5765 (33)	−171 (27)	30 (7)
O(10)	3640 (31)	4994 (28)	−772 (22)	48 (7)
C(11)	5032 (44)	8482 (39)	508 (31)	42 (9)
O(11)	6141 (34)	9162 (29)	339 (23)	52 (7)
C(12)	1835 (41)	7674 (33)	119 (30)	37 (8)
O(12)	1016 (37)	7900 (34)	−597 (27)	64 (8)
C(13)	1917 (40)	5389 (35)	4550 (29)	36 (8)
O(13)	1750 (31)	5822 (28)	5404 (23)	49 (7)
C(14)	2658 (42)	4049 (38)	1785 (30)	38 (8)
O(14)	2984 (38)	3561 (34)	1039 (27)	66 (9)
C(15)	677 (39)	3101 (35)	3156 (28)	34 (8)
O(15)	−372 (37)	2141 (32)	3066 (25)	61 (8)
C(16)	3795 (38)	3961 (34)	3761 (27)	31 (7)
O(16)	4642 (40)	3470 (35)	4035 (28)	70 (9)

Table 2. Bond lengths (Å)

Table 2. *Bond lengths* (Å)

Os(1)—Os(2)	2·796 (5)	Os(1)—Os(3)	2·845 (5)
Os(1)—Os(4)	2·969 (5)	Os(1)—Os(5)	2·893 (5)
Os(2)—Os(3)	2·843 (5)	Os(2)—Os(4)	2·955 (5)
Os(2)—Os(5)	2·893 (5)	Os(3)—Os(4)	
Os(1)—C(1)	1·849 (41)	C(1)—O(1)	1·238 (49)
Os(1)—C(2)	1·909 (49)	C(2)—O(2)	1·134 (58)
Os(1)—C(3)	1·834 (33)	C(3)—O(3)	1·185 (41)
Os(2)—C(4)	1·878 (38)	C(4)—O(4)	1·130 (47)
Os(2)—C(5)	1·904 (34)	C(5)—O(5)	1·156 (43)
Os(2)—C(6)	1·889 (48)	C(6)—O(6)	1·169 (61)
Os(3)—C(7)	1·920 (41)	C(7)—O(7)	1·068 (52)
Os(3)—C(8)	1·859 (38)	C(8)—O(8)	1·131 (49)
Os(3)—C(9)	1·847 (61)	C(9)—O(9)	1·180 (72)
Os(4)—C(10)	1·937 (36)	C(10)—O(10)	1·096 (45)
Os(4)—C(11)	1·953 (39)	C(11)—O(11)	1·099 (47)
Os(4)—C(12)	1·826 (45)	C(12)—O(12)	1·298 (58)
Os(5)—C(13)	1·917 (39)	C(13)—O(13)	1·176 (48)
Os(5)—C(14)	1·924 (41)	C(14)—O(14)	1·117 (53)
Os(5)—C(15)	1·906 (33)	C(15)—O(15)	1·158 (43)
Os(5)—C(16)	1·908 (42)	C(16)—O(16)	1·130 (59)

Table 3. Bond angles (°)

Table 3. *Bond angles* (°)

Os(2)—Os(1)—Os(3)	60·5 (1)	Os(2)—Os(1)—Os(4)	61·6 (1)
Os(3)—Os(1)—Os(4)	57·3 (1)	Os(2)—Os(1)—Os(5)	61·1 (1)
Os(3)—Os(1)—Os(5)	121·6 (2)	Os(4)—Os(1)—Os(5)	95·2 (2)
Os(1)—Os(2)—Os(3)	60·6 (1)	Os(1)—Os(2)—Os(4)	62·1 (1)
Os(3)—Os(2)—Os(4)	57·4 (1)	Os(1)—Os(2)—Os(5)	61·1 (1)
Os(3)—Os(2)—Os(5)	121·7 (2)	Os(·)—Os(2)—Os(5)	95·4 (1)
Os(1)—Os(3)—Os(2)	58·9 (1)	Os(1)—Os(3)—Os(4)	63·6 (1)
Os(2)—Os(3)—Os(4)	63·3 (2)	Os(1)—Os(4)—Os(2)	56·3 (1)
Os(1)—Os(4)—Os(3)	59·1 (1)	Os(2)—Os(4)—Os(3)	59·3 (1)
Os(1)—Os(5)—Os(2)	57·8 (1)		
Os(1)—C(1)—O(1)	175·7 (32)	Os(1)—C(2)—O(2)	168·0 (31)
Os(1)—C(3)—O(3)	174·8 (36)	Os(2)—C(4)—O(4)	176·6 (35)
Os(2)—C(5)—O(5)	168·0 (39)	Os(2)—C(6)—O(6)	169·8 (35)
Os(3)—C(7)—O(7)	175·1 (40)	Os(3)—C(8)—O(8)	176·6 (40)
Os(3)—C(9)—O(9)	171·0 (37)	Os(4)—C(10)—O(10)	176·6 (30)
Os(4)—C(11)—O(11)	174·0 (42)	Os(4)—C(12)—O(12)	167·2 (33)
Os(5)—C(13)—O(13)	175·5 (32)	Os(5)—C(14)—O(14)	168·0 (42)
Os(5)—C(15)—O(15)	175·1 (35)	Os(5)·C(16)—O(16)	175·2 (29)
Os(2)—Os(1)—C(1)	97·4 (12)	Os(3)·-Os(1)—C(1)	92·0 (12)
Os(4)—Os(1)—C(1)	148·1 (10)	Os(5)—Os(1)—C(1)	94·3 (15)
Os(2)—Os(1)—C(2)	133·4 (11)	Os(3)—Os(1)—C(2)	73·1 (11)
Os(4)—Os(1)—C(2)	90·4 (13)	Os(5)—Os(1)—C(2)	164·8 (10)
C(1)—Os(1)—C(2)	88·3 (19)	Os(2)-Os(1)—C(3)	137·2 (13)
Os(3)—Os(1)—C(3)	160·8 (13)	Os(4)—Os(1)—C(3)	119·7 (12)
Os(5)—Os(1)—C(3)	76·7 (14)	C(1)-Os(1)—C(3)	92·2 (16)
C(2)—Os(1)—C(3)	88·3 (18)	Os(1)—Os(2)—C(4)	93·9 (11)
Os(3)—Os(2)—C(4)	89·6 (14)	Os(4)—Os(2)—C(4)	145·3 (13)
Os(5)—Os(2)—C(4)	93·5 (11)	Os(1)—Os(2)—C(5)	134·3 (13)
Os(3)—Os(2)—C(5)	74·1 (14)	Os(4)—Os(2)—C(5)	89·5 (12)
Os(5)—Os(2)—C(5)	163·4 (13)	C(4)—Os(2)—C(5)	91·4 (15)
Os(1)—Os(2)—C(6)	135·9 (10)	Os(3)—Os(2)—C(6)	162·9 (11)
Os(4)—Os(2)—C(6)	121·7 (13)	Os(5)—Os(2)—C(6)	75·0 (12)
C(4)—Os(2)—C(6)	93·0 (18)	C(5)—Os(2)—C(6)	88·9 (18)
Os(1)—Os(3)—C(7)	97·6 (12)	Os(2)-Os(3)—C(7)	99·1 (15)
Os(4)—Os(3)—C(7)	158·7 (13)	Os(1)—Os(3)—C(8)	161·0 (13)
Os(2)—Os(3)—C(8)	106·9 (15)	Os(4)—Os(3)—C(8)	99·5 (13)
C(7)—Os(3)—C(8)	97·2 (17)	Os(1)—Os(3)-C(9)	109·0 (14)
Os(2)—Os(3)—C(9)	163·9 (14)	Os(4)—Os(3)—C(9)	102·6 (16)
C(7)—Os(3)—C(9)	92·8 (22)	C(8)-Os(3)—C(9)	82·1 (21)
Os(1)—Os(4)-C(10)	121·0 (11)	Os(2)-Os(4)—C(10)	120·8 (12)
Os(3)—Os(4) ·C(10)	180·0 (15)	Os(1)—Os(4)—C(11)	144·8 (11)
Os(2)—Os(4)—C(11)	98·2 (13)	Os(3)—Os(4)—C(11)	87·7 (13)
C(10)—Os(4)—C(11)	92·2 (16)	Os(1)—Os(4)—C(12)	94·2 (13)
Os(2)·-Os(4)—C(12)	141·3 (12)	Os(3)—Os(4)—C(12)	84·9 (13)
C(10)—Os(4)—C(12)	95·1 (17)	C(11)—Os(4)—C(12)	94·2 (18)
Os(1)—Os(5)—C(13)	86·7 (14)	Os(2)—Os(5)—C(13)	88·6 (11)
Os(1)—Os(5)—C(14)	88·9 (14)	Os(2)—Os(5)—C(14)	90·7 (12)
C(13)—Os(5)—C(14)	175·3 (18)	Os(1)—Os(5)—C(15)	106·0 (13)
Os(2)—Os(5)—C(15)	163·8 (13)	C(13)—Os(5)—C(15)	90·7 (15)
C(14)—Os(5)—C(15)	88·7 (16)	Os(1)—Os(5)—C(16)	161·1 (9)
Os(2)—Os(5)—C(16)	103·7 (10)	C(13)—Os(5)—C(16)	88·8 (17)
C(14)—Os(5)—C(16)	95·9 (18)	C(15)·-Os(5)—C(16)	92·5 (16)

tropic temperature factors for Os, and isotropic for C and O. The weighting scheme $w = [\sigma^2(F) + 0.003F^2]^{-1}$ led to mean values of $w\Delta^2$ which showed little systematic variation with the magnitude of F_o or $\sin \theta / \lambda$. Positional and isotropic thermal parameters are given in Table 1, bond lengths and angles in Tables 2 and 3. Fig. 1 shows the atom labelling.*

Discussion. Although no crystallographic symmetry is imposed on the molecule, it possesses approximate C_s symmetry with the mirror plane running through Os(3), Os(4) and Os(5). Os(1), Os(2), Os(3) and Os(5) are coplanar to within 0·01 Å, with Os(4) 2·39 Å above this plane. The Os—C and C—O lengths are effectively equal, with mean values of 1·89 and 1·15 Å. There are no Os$\cdots$C non-bonded contacts $<2\cdot93$ Å [Os(3)$\cdots$C(2)], so there are no incipient bridging carbonyls as found, for example, in $Os_5(CO)_{16}$ (Reichert & Sheldrick, 1977). NMR measurements by Eady, Johnson & Lewis (1977) indicate that there are two equivalent edge-bridging hydrides; from the disposition of the carbonyl ligands they probably bridge the two long Os—Os bonds [Os(1)—Os(4) and Os(2)—Os(4)]. If the two Os—H—Os bridges are considered to be two-electron three-centre bonds, a structure based on a square pyramid of Os atoms obeys the 18-electron rule overall, and would probably have

been predicted on the basis of Wade's theory. The less symmetrical structure actually adopted makes the same number of bonds, and so also obeys a net 18-electron rule.

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1-(1-Naphthyl)isoquinoline

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Abstract. $C_{19}H_{13}N$, triclinic, $P\bar{1}$, $Z = 4$, $M_r = 255\cdot3$, $a = 11\cdot363$ (3), $b = 12\cdot243$ (3), $c = 11\cdot305$ (3) Å, $\alpha = 100\cdot87$ (2), $\beta = 105\cdot38$ (2), $\gamma = 108\cdot46$ (2)°, $D_x = 1\cdot285$, $D_m = 1\cdot23$ g cm^{-3}, μ(Cu) $= 5\cdot6$ cm^{-1}, final $R = 0\cdot040$. The sp^2–sp^2 bond connecting the two halves is 1·501 (3) Å in one of the molecules and 1·495 (3) Å in the other. The planar halves of each molecule are inclined at 77·5 and 63·9°.

Introduction. The present study was undertaken in connection with an investigation of the steric effect of N atom lone-pair electrons performed by Dr J. Pedersen (Department of Organic Chemistry, Chalmers University of Technology). A crystal 0·3 × 0·4 × 0·5 mm was used for the data collection. Cell dimensions and

intensities were measured on a Syntex $P2_1$ automatic four-circle diffractometer with graphite-monochromated Cu $K\alpha$ radiation. The θ–2θ scan procedure was used and the 2θ scan speed was allowed to vary between 1·5 and 8° min^{-1} depending on the intensity of the reflection. Each reflection was measured in 96 steps and the intensities were evaluated by a profile-analysis procedure (Lehmann & Larsen, 1974). Of the 3449 independent reflections with $2\theta \leq 115°$, 2572 had $I > 3\sigma(I)$ and were considered observed. A control reflection monitored every 50 reflections showed no significant fluctuation.

The structure was solved with the symbolic addition program system *SIMPEL* (Overbeek, van der Putten & Schenk, 1976). Triplet and quartet relations were