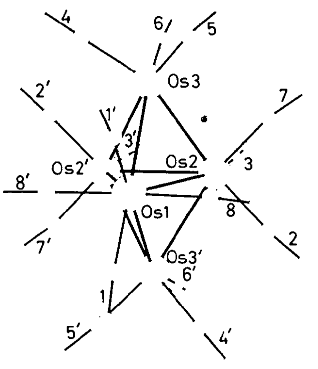

Fig. 1. Molecular structure and numbering scheme.

each bonded to three carbonyls. The significantly longer Os—Os bonds to Os(1) may serve to redress the resulting electron imbalance. The Os—C and C—O bonds are respectively equal within experimental error, with means 1·89 (4) and 1·16 (4) Å; refinement with the C—O distances constrained to be equal yielded 1·16 (1) Å. There are two short non-bonded Os···C interactions, both associated with significant departures from linear Os—C—O units: C(8)···Os(2) 2·66 (4), C(7)···Os(3) 2·89 (4) Å; Os(1)—C(8)—O(8) 166 (3), Os(2)—C(7)—O(7) 171 (4)°. The molecular structure (Fig. 1) is consistent with the Johnson (1976) model in which close-packed carbonyl groups adopt a polyhedral arrangement. The high density [3·83 g cm^{-3}, in contrast to 3·19 g cm^{-3} in $Os_6(CO)_{18}$; Mason, Thomas & Mingos, 1973] implies efficient packing of the molecules, which can be seen in Fig. 2.

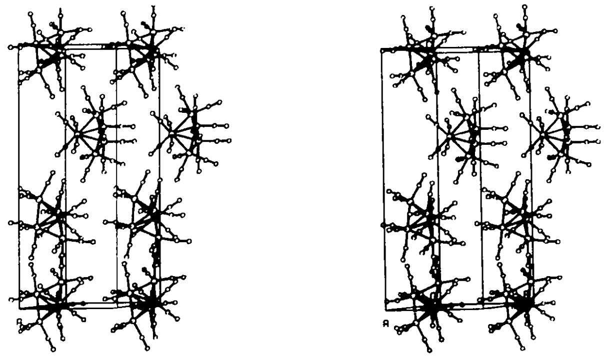

Fig. 2. Stereoscopic view of the crystal packing down **a***.

We are grateful to the Science Research Council for providing the diffractometer, to ICI for a Fellowship (to BER), and to Dr C. R. Eady for providing the crystals. Calculations were performed with the Cambridge University IBM 370/165 computer and programs written by BER and GMS; the figures were drawn with *PLUTO* written by Dr W. D. S. Motherwell.

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Acta Cryst. (1977). B**33**, 175–177

Di-μ-cyclononaallyl-hexacarbonyldiosmium

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(*Received* 23 *July* 1976; *accepted* 7 *August* 1976)

Abstract. $C_{24}H_{28}O_6Os_2$, monoclinic, $P2_1/c$, $a = 7{\cdot}174\,(4)$, $b = 8{\cdot}850\,(4)$, $c = 19{\cdot}173\,(10)$ Å, $\beta = 103{\cdot}11\,(4)°$, $U = 1185{\cdot}6$ Å^3, $Z = 2$, $D_x = 2{\cdot}22$ g cm^{-3}. The structure was solved by the heavy-atom method and refined to an R of 0·027 for 1145 independent diffractometer data. The centrosymmetric molecule contains two $Os(CO)_3$ units, with each Os σ-bonded to the central allyl C of a cyclononaallyl group π-bonded to the other Os.

Introduction. When $Fe_2(CO)_9$ reacts with cyclonona-1,2-diene, the latter dimerizes and the product contains a di(cyclononaallyl) group π-bonded to an $Fe_2(CO)_6$ unit which retains an Fe—Fe bond (Howell, Lewis, Matheson & Russell, 1975). The same diene reacts with $H_2Os_3(CO)_{10}$ to yield a product of similar empirical formula (with Fe replaced by Os; Bryan, Johnson & Lewis, 1976), but the single-crystal X-ray determination reported here reveals a quite different structure.

Intensities were determined with an automated Stoe STADI-2 two-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation, and a crystal 0·07 × 0·10 × 0·17 mm (layers 0–4kl). An inferior crystal mounted about [010] was used only for cell-constant determination, which was based on a least-squares fit to diffractometer ω angles for the 0kl and h0l zero-layer reflexions. Lp and numerical absorption corrections were applied ($\mu = 103{\cdot}4$ cm^{-1}); equivalent reflexions were averaged to give 1145 unique reflexions with $F > 2\sigma(F)$.

The Os coordinates were derived from the Patterson map and the C and O atoms located in difference syntheses; no attempt was made to locate the H atoms. The structure was refined by full-matrix least squares with complex neutral-atom scattering factors and the weighting scheme $w = 1/[\sigma^2(F) + 0{\cdot}00126|F_o|^2]$. The parameters included anisotropic temperature factors for Os only, interlayer scale factors, and an empirical extinction parameter x which refined to 0·00020 (8); F_c is multiplied by $(1 - 0{\cdot}0001xF_c^2/\sin\theta)$. The constraint $U_{11} = (U_{22} + U_{33})/2$ was applied to reduce correlation involving the interlayer scale factors. The refinement converged to $R' = \Sigma\, w^{1/2}\Delta/\Sigma\, w^{1/2}\, F_o = 0{\cdot}029$ and $R = 0{\cdot}027$. Final coordinates and thermal parameters are given in Tables 1 and 2, the resulting bond lengths and angles in Table 3.*

Table 1. *Atom coordinates* (×10^4) *and isotropic temperature factors* (Å^2 ×10^3)

	x	y	z	U
Os(1)	316 (1)	2002	194	
C(1)	1505 (19)	3428 (11)	917 (6)	31 (2)
O(1)	2253 (14)	4329 (8)	1317 (5)	50 (2)
C(2)	−2089 (21)	3002 (11)	47 (6)	34 (2)
O(2)	−3599 (17)	3509 (11)	−41 (6)	66 (3)
C(3)	1315 (19)	3018 (10)	−529 (5)	32 (2)
O(3)	1835 (13)	3594 (4)	−974 (4)	47 (2)
C(4)	−950 (18)	367 (9)	−611 (5)	22 (2)
C(5)	291 (18)	−336 (9)	−1033 (5)	27 (2)
C(6)	−361 (18)	−848 (11)	−1814 (5)	33 (2)
C(7)	−817 (21)	509 (11)	−2333 (6)	39 (3)
C(8)	−2955 (22)	912 (14)	−2547 (7)	55 (3)
C(9)	−3761 (21)	1647 (13)	−1957 (7)	52 (3)
C(10)	−5174 (21)	718 (12)	−1624 (7)	47 (3)
C(11)	−4278 (19)	−729 (10)	−1260 (6)	35 (2)
C(12)	−2781 (18)	−334 (10)	−582 (5)	30 (2)

Table 2. *Anisotropic temperature factors* (Å^2 ×10^3)

The temperature factor exponent takes the form: $-2\pi^2(U_{11}h^2a^{*2} + \cdots + 2U_{12}hka^*b^*)$.

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Os(1)	27 (1)	24 (1)	30 (1)	0 (1)	4 (1)	1 (1)

Table 3. *Bond lengths* (Å) *and angles* (°)

C(1)–Os(1)	1·92 (1)	C(7)–C(6)	1·55 (1)
C(2)–Os(1)	1·90 (1)	C(8)–C(7)	1·54 (2)
C(3)–Os(1)	1·92 (1)	C(9)–C(8)	1·53 (2)
O(1)–C(1)	1·15 (1)	C(10)–C(9)	1·55 (2)
O(2)–C(2)	1·15 (2)	C(11)–C(10)	1·53 (2)
O(3)–C(3)	1·13 (1)	C(12)–C(11)	1·53 (2)
C(4)–Os(1)	2·16 (1)	C(4)–Os(1′)	2·25 (1)
C(5)–C(4)	1·47 (2)	C(5)–Os(1′)	2·29 (1)
C(12)–C(4)	1·46 (2)	C(12)–Os(1′)	2·29 (1)
C(6)–C(5)	1·53 (1)	Os(1)–Os(1′)	3·629 (3)
C(2)–Os(1)–C(1)	92·3 (5)	C(7)–C(6)–C(5)	111·9 (8)
C(3)–Os(1)–C(1)	92·1 (4)	C(8)–C(7)–C(6)	113·9 (10)
C(3)–Os(1)–C(2)	98·6 (5)	C(9)–C(8)–C(7)	115·1 (11)
O(1)–C(1)–Os(1)	175·8 (9)	C(10)–C(9)–C(8)	118·1 (10)
O(2)–C(2)–Os(1)	175·2 (10)	C(11)–C(10)–C(9)	112·7 (11)
O(3)–C(3)–Os(1)	177·1 (10)	C(12)–C(11)–C(10)	109·8 (8)
C(12)–C(4)–C(5)	120·1 (8)	C(11)–C(12)–C(4)	121·9 (9)
C(6)–C(5)–C(4)	125·1 (10)		

Discussion. The molecule (Fig. 1) possesses a crystallographic centre of symmetry. The Os···Os distance of 3·629 (3) Å indicates negligible Os–Os interaction. The allyl group dimensions are typical of π-allylic systems (Gatehouse, Reichert & West, 1974, and references therein), and the distance of Os(1) from C(4) (the central allyl carbon) of 2·16 (1) Å is consistent with a σ bond (see *e.g.* Reichert & Sheldrick, 1977). This structure is clearly a possible intermediate in the ligand dimerization reaction of the iron analogue.

We are grateful to Mr E. G. Bryan for providing the crystals, to ICI for financial support to BER, and to the Science Research Council for the diffractometer. The calculations were performed on the Cambridge University IBM 370/165 computer with programs written by GMS. Fig. 1 was drawn with *PLUTO* written by Dr W. D. S. Motherwell.

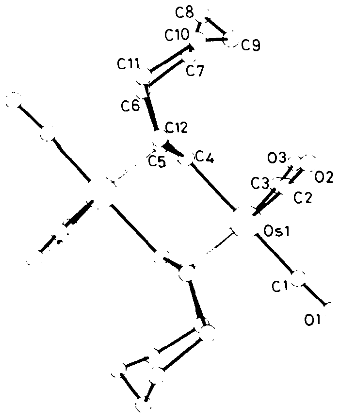

Fig. 1. The molecule of di-μ-cyclononaallyl-hexacarbonyldiosmium with unique atoms labelled.

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32009 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

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Acta Cryst. (1977). B**33**, 177–179

20-Methyl-14β,17α-pregn-4-en-3-one

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(*Received* 2 *August* 1976; *accepted* 14 *August* 1976)

Abstract. $C_{22}H_{34}O$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}950\,(2)$, $b = 32{\cdot}237\,(10)$, $c = 7{\cdot}281\,(1)$ Å, $M_r = 314{\cdot}5$, $Z = 4$, $D_x = 1{\cdot}12$ g cm^{-3}. The ring junction *C*/*D* is *cis*, while ring junctions *A*/*B* and *B*/*C* are both *trans*. Ring conformations are: *A* 1α,2β half chair; *B*,*C* chair; *D* 14β envelope.

Introduction. Cell dimensions were obtained from a least-squares fit to the settings of 15 reflexions ($\pm hkl$) on a Syntex $P2_1$ diffractometer (Mo $K\alpha$ 0·71069 Å). Intensity measurements were carried out in the θ–$2\theta'$ mode ($3{\cdot}0 \le 2\theta \le 50{\cdot}0°$) with graphite-monochromated Mo $K\alpha$ radiation, at scan speeds varying linearly between 2·93° min^{-1} (150 c.p.s. and below) and 19·53° min^{-1} (5000 c.p.s. and above). Scan and background times were equal. Lorentz and polarization but no absorption [μ(Mo $K\alpha$) = 0·34 cm^{-1}] corrections were applied. After application of the acceptance criterion $I \ge 1{\cdot}5\sigma(I)$, 1040 unique

Table 1. *Atom positional parameters* ($\times 10^4$) *and anisotropic temperature factors* (Å^2 $\times 10^3$)

$$T = \exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{23}klb^*c^* + 2U_{13}lhc^*a^* + 2U_{12}hka^*b^*)].$$

	x	y	z	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1)	−128 (10)	2337 (2)	6717 (10)	62 (5)	48 (5)	66 (6)	1 (5)	3 (5)	−5 (5)
C(2)	761 (12)	2727 (3)	7566 (16)	73 (7)	58 (7)	145 (10)	33 (7)	−22 (8)	−2 (5)
C(3)	782 (10)	2699 (3)	9639 (17)	44 (6)	50 (6)	137 (11)	−18 (8)	−8 (7)	−7 (5)
C(3)	684 (8)	3010 (2)	589 (10)	103 (5)	70 (4)	166 (7)	−59 (5)	6 (6)	−9 (4)
C(4)	999 (10)	2289 (3)	387 (11)	74 (6)	99 (8)	42 (6)	−13 (6)	7 (5)	−30 (6)
C(5)	1052 (10)	1939 (3)	9390 (12)	46 (5)	59 (6)	56 (6)	−10 (5)	6 (5)	−18 (5)
C(6)	1563 (13)	1521 (3)	176 (10)	144 (9)	80 (6)	46 (6)	10 (5)	−44 (6)	−36 (7)
C(7)	160 (12)	1206 (2)	9918 (11)	93 (7)	53 (5)	58 (6)	14 (5)	−4 (6)	−20 (5)
C(8)	−265 (10)	1158 (2)	7886 (9)	48 (5)	48 (5)	35 (5)	17 (4)	−7 (4)	3 (4)
C(9)	−669 (8)	1574 (2)	7008 (9)	41 (5)	41 (4)	27 (4)	−1 (4)	7 (4)	1 (4)
C(10)	665 (9)	1926 (2)	7350 (10)	55 (5)	36 (5)	40 (5)	7 (4)	11 (5)	0 (4)
C(11)	−1061 (10)	1500 (2)	4965 (9)	53 (5)	48 (5)	41 (5)	11 (4)	5 (5)	−12 (4)
C(12)	−2539 (10)	1210 (2)	4716 (9)	55 (5)	57 (5)	31 (5)	4 (4)	−2 (4)	−3 (4)
C(13)	−2274 (8)	774 (2)	5620 (10)	31 (4)	43 (5)	42 (5)	−10 (4)	10 (4)	6 (4)
C(14)	−1679 (10)	844 (2)	7618 (10)	69 (5)	23 (4)	40 (5)	9 (4)	−12 (5)	−1 (4)
C(15)	−3317 (10)	929 (2)	8691 (10)	55 (5)	82 (6)	31 (5)	−4 (4)	27 (5)	−23 (5)
C(16)	−4726 (9)	727 (3)	7563 (12)	45 (5)	80 (6)	73 (6)	−5 (6)	−2 (5)	−8 (5)
C(17)	−3901 (8)	528 (2)	5904 (10)	39 (4)	39 (4)	48 (5)	−3 (4)	9 (4)	0 (4)
C(18)	−1014 (12)	529 (3)	4503 (14)	55 (6)	70 (6)	55 (6)	1 (6)	1 (6)	−10 (5)
C(19)	2313 (13)	1841 (3)	6288 (11)	57 (6)	81 (7)	53 (6)	1 (6)	10 (6)	−4 (5)
C(20)	−5029 (9)	420 (3)	4255 (11)	40 (5)	65 (6)	45 (6)	−5 (5)	9 (5)	−7 (5)
C(21)	−6260 (11)	70 (3)	4794 (16)	59 (7)	57 (6)	104 (10)	7 (6)	−33 (7)	−20 (5)
C(22)	−6030 (12)	772 (3)	3349 (14)	68 (7)	81 (7)	71 (7)	14 (6)	−21 (6)	−12 (6)