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μ-But-1-enyl-μ-hydrido-decacarbonyl-*triangulo*-triosmium

BY JOHN J. GUY, BERNHARD E. REICHERT AND GEORGE M. SHELDRICK
University Chemical Laboratory, Lensfield Road, Cambridge CB2 1EW, England

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Abstract. $C_{14}H_8O_{10}Os_3$, monoclinic, $C2/c$, $a=20{\cdot}30$ (1), $b=15{\cdot}54$ (1), $c=18{\cdot}19$ (2) Å, $\beta=137{\cdot}18$ (3)°, $U=3899$ Å^3, $Z=8$, $D_x=3{\cdot}09$ g cm^{-3}. The structure was solved by heavy-atom methods and refined to an R of 0·032 for 1415 unique two-circle diffractometer data. A partially disordered but-1-enyl group asymmetrically bridges one edge of the Os_3 triangle. All the carbonyl groups are terminal, with four attached to the Os atom furthest from the alkene and three to each of the remaining Os atoms.

Introduction. The structure of the title compound has been determined by single-crystal X-ray diffraction in order to confirm the structure proposed on chemical and spectroscopic grounds (Jackson, Johnson, Kelland, Lewis & Schorpp, 1975).

Intensities were determined with an automated Stoe two-circle diffractometer, Mo $K\alpha$ radiation, graphite monochromator, and a crystal $0{\cdot}12\times0{\cdot}08\times0{\cdot}06$ mm (layers $hk0$–$hk17$). Data were also collected for a crystal mounted about [010], but since the reflexion profiles were poor these were only used for cell-constant determination. The cell dimensions were found by a least-squares fit to the diffractometer ω angle measurements for all the $hk0$ and $h0l$ zero-layer reflexions ($\lambda=0{\cdot}71069$ Å). Lp and empirical absorption corrections were applied (with an azimuthal scan of the 006 reflexion after realignment of the crystal so that c^* was along the oscillation axis).

The three Os atoms were located by multisolution $\sum_2$ sign expansion, and the light atoms by successive weighted difference syntheses. The structure was refined by full-matrix least squares with anisotropic Os and isotropic light atoms. No attempt was made to locate the bridging hydride or ligand H atoms. The ethyl tail of the bridging alkene was found to be disordered, the site occupation factors of the two alternative conformations refining to 0·43 (8) [C(13′), C(14′)] and 0·57 (8) [C(13), C(14)] when their sum was constrained to be one. Chemically equivalent C···C distances involving the ethyl group were also constrained to be equal. Interlayer scale factors were allowed to refine to correct for the μ dependence of the empirical absorption corrections. Complex neutral atom scattering factors were employed, with the weighting scheme $w=1{\cdot}76/[\sigma^2(F_o)+0{\cdot}00006|F_o|^2]$. The final $R'=\sum w^{1/2}\Delta/\sum w^{1/2}|F_o|$ was 0·032, with a corresponding R of 0·032. Final atomic coordinates and temperature factors are presented in Tables 1 and 2. The constraint $U_{33}=\frac{1}{2}(U_{11}+U_{22})$ was applied to the anisotropic temperature factors to reduce correlation with the interlayer scale factors. A final difference synthesis revealed no features greater than 0·9 e Å^{-3}.*

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31927 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Atom coordinates* ($\times10^4$) *and isotropic temperature factors* (Å$^2\times10^3$)

	x	y	z	U
Os(1)	2347 (1)	3866 (1)	3606 (1)	
Os(2)	1019 (1)	3702 (1)	1321 (1)	
Os(3)	2738 (1)	4664 (1)	2502 (1)	
C(1)	3461 (15)	4225 (13)	5031 (19)	53 (6)
O(1)	4132 (12)	4453 (10)	5917 (14)	76 (5)
C(2)	1694 (15)	3213 (13)	3770 (18)	55 (6)
O(2)	1293 (11)	2788 (11)	3871 (13)	73 (5)
C(3)	3008 (13)	2847 (13)	3802 (17)	48 (5)
O(3)	3396 (11)	2213 (11)	3958 (14)	78 (5)
C(4)	736 (16)	2589 (14)	1393 (19)	59 (6)
O(4)	535 (11)	1909 (10)	1436 (14)	78 (5)
C(5)	4060 (17)	4402 (15)	3681 (20)	64 (6)
O(5)	4858 (14)	4194 (11)	4408 (16)	95 (6)
C(6)	1572 (13)	4855 (12)	3255 (16)	46 (5)
O(6)	1169 (11)	5438 (10)	3118 (13)	69 (4)
C(7)	295 (17)	3608 (15)	−116 (22)	71 (7)
O(7)	−163 (13)	3563 (11)	−1055 (17)	92 (6)
C(8)	3 (16)	4234 (12)	1046 (18)	51 (5)
O(8)	−638 (11)	4525 (10)	838 (13)	72 (5)
C(9)	2842 (14)	5610 (14)	3227 (17)	46 (5)
O(9)	2904 (11)	6221 (10)	3638 (14)	74 (5)
C(10)	2846 (15)	5386 (14)	1779 (20)	57 (6)
O(10)	2902 (12)	5864 (11)	1322 (15)	82 (5)
C(11)	2281 (13)	3332 (12)	1734 (16)	42 (5)
C(12)	2536 (19)	3702 (17)	1270 (23)	81 (8)
C(13)	3519 (31)	3517 (44)	1717 (49)	92 (23)
C(14)	3446 (52)	2875 (46)	1075 (57)	150 (34)
C(13′)	3384 (50)	3164 (49)	1674 (72)	104 (36)
C(14′)	3756 (60)	3720 (51)	1397 (73)	125 (39)

Table 2. *Anisotropic temperature factors* (Å$^2\times10^3$)

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

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Os(1)	43	46	45	6	31	2
Os(2)	41	48	44	0	27	−5
Os(3)	42	53	47	1	32	−5

Table 3. *Bond lengths* (Å)

Os(2)–Os(1)	2·858 (1)	O(1)—C(1)	1·16 (2)
Os(3)–Os(1)	2·923 (1)	O(2)—C(2)	1·16 (2)
Os(3)–Os(2)	2·834 (1)	O(3)—C(3)	1·16 (2)
C(1)—Os(1)	1·88 (2)	O(4)—C(4)	1·16 (3)
C(2)—Os(1)	1·86 (2)	O(5)—C(5)	1·15 (3)
C(3)—Os(1)	1·93 (2)	O(6)—C(6)	1·12 (2)
C(6)—Os(1)	1·94 (2)	O(7)—C(7)	1·21 (3)
C(4)—Os(2)	1·86 (2)	O(8)—C(8)	1·15 (2)
C(7)—Os(2)	1·84 (3)	O(9)—C(9)	1·16 (2)
C(8)—Os(2)	1·92 (2)	O(10)–C(10)	1·18 (3)
C(5)—Os(3)	1·88 (2)	C(12)–C(11)	1·40 (3)
C(9)—Os(3)	1·88 (2)	C(13)–C(12)	1·53 (4)
C(10)–Os(3)	1·86 (3)	C(14)–C(13)	1·46 (6)
C(11)–Os(2)	2·15 (2)	C(13′)–C(12)	1·53 (4)
C(11)–Os(3)	2·28 (2)	C(14′)–C(13′)	1·46 (6)
C(12)–Os(3)	2·46 (3)		

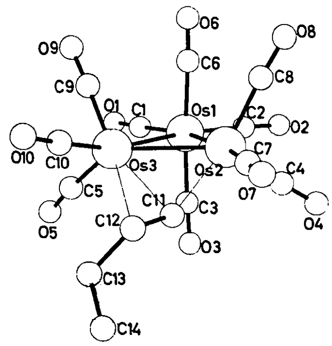

Fig. 1. The molecule of μ-but-1-enyl-μ-hydrido-decarbonyl-*triangulo*-triosmium.

Table 4. *Bond angles* (°)

Os(3)–Os(1)–Os(2)	58·7 (0)	C(9)—Os(3)–C(5)	92·2 (10)
Os(3)–Os(2)–Os(1)	61·8 (0)	C(10)–Os(3)–Os(1)	167·0 (7)
Os(2)–Os(3)–Os(1)	59·5 (0)	C(10)–Os(3)–Os(2)	120·0 (8)
C(1)—Os(1)–Os(2)	158·0 (7)	C(10)–Os(3)–C(5)	97·4 (11)
C(1)—Os(1)–Os(3)	99·3 (7)	C(10)–Os(3)–C(9)	90·9 (10)
C(2)—Os(1)–Os(2)	99·3 (8)	C(11)–Os(3)–Os(1)	80·9 (8)
C(2)—Os(1)–Os(3)	158·0 (7)	C(11)–Os(3)–Os(2)	48·3 (5)
C(2)—Os(1)–C(1)	102·6 (11)	C(11)–Os(3)–C(5)	95·8 (9)
C(3)—Os(1)–Os(2)	89·0 (7)	C(11)–Os(3)–C(9)	157·7 (8)
C(3)—Os(1)–Os(3)	88·5 (7)	C(11)–Os(3)–C(10)	108·7 (10)
C(3)—Os(1)–C(1)	91·1 (9)	C(12)–Os(3)–Os(1)	114·8 (8)
C(3)—Os(1)–C(2)	90·2 (10)	C(12)–Os(3)–Os(2)	72·5 (7)
C(6)—Os(1)–Os(2)	85·4 (7)	C(12)–Os(3)–C(5)	93·0 (11)
C(6)—Os(1)–Os(3)	90·1 (7)	C(12)–Os(3)–C(9)	166·0 (9)
C(6)—Os(1)–C(1)	94·8 (9)	C(12)–Os(3)–C(10)	75·5 (10)
C(6)—Os(1)–C(2)	89·0 (9)	C(12)–Os(3)–C(11)	34·0 (7)
C(6)—Os(1)–C(3)	174·1 (8)	O(1)—C(1)—Os(1)	177·8 (20)
C(4)—Os(2)–Os(1)	89·5 (8)	O(2)—C(2)—Os(1)	178·5 (19)
C(4)—Os(2)–Os(3)	135·3 (7)	O(3)—C(3)—Os(1)	175·6 (20)
C(7)—Os(2)–Os(1)	173·0 (7)	O(4)—C(4)—Os(2)	177·4 (21)
C(7)—Os(2)–Os(3)	112·1 (8)	O(5)—C(5)—Os(3)	176·1 (21)
C(7)—Os(2)–C(4)	93·4 (11)	O(6)—C(6)—Os(1)	175·8 (18)
C(8)—Os(2)–Os(1)	91·6 (8)	O(7)—C(7)—Os(2)	178·1 (21)
C(8)—Os(2)–Os(3)	117·5 (7)	O(8)—C(8)—Os(2)	176·6 (19)
C(8)—Os(2)–C(4)	95·2 (10)	O(9)—C(9)—Os(3)	176·2 (20)
C(8)—Os(2)–C(7)	94·5 (11)	O(10)–C(10)–Os(3)	177·9 (20)
C(11)–Os(2)–Os(1)	84·5 (7)	C(12)–C(11)–Os(2)	123·6 (17)
C(11)–Os(2)–Os(3)	52·3 (6)	C(12)–C(11)–Os(3)	80·3 (15)
C(11)–Os(2)–C(4)	94·4 (10)	C(11)–C(12)–Os(3)	65·8 (14)
C(11)–Os(2)–C(7)	88·9 (10)	C(13)–C(12)–C(11)	121·2 (36)
C(11)–Os(2)–C(8)	169·6 (7)	C(14)–C(13)–C(12)	112·1 (48)
C(5)—Os(3)–Os(1)	90·2 (9)	C(13′)–C(12)–C(11)	105·7 (34)
C(5)—Os(3)–Os(2)	133·2 (7)	C(14′)–C(13′)–C(12)	101·2 (53)
C(9)—Os(3)–Os(1)	78·3 (8)	C(13′)–C(12)–C(13)	22·3 (37)
C(9)—Os(3)–Os(2)	112·7 (7)		

Discussion. The title compound is obtained from the reaction of $H_2Os_3(CO)_{10}$ with but-1-yne (Jackson *et al.*, 1975); the structure found here (Fig. 1) is essentially that originally proposed. Only one orientation of the disordered ethyl group is shown for clarity. A hydride bridging the Os(2)–Os(3) edge was proposed on the basis of spectroscopic evidence, but could not be unambiguously located from the X-ray data.

Bond lengths and angles are given in Tables 3 and 4. The difference in the angles C(13)–C(12)–C(11) and C(13′)–C(12)–C(11) is probably caused by slightly different positions for C(12) associated with the two possible conformations of the disordered ethyl group. The metal–carbon distances suggest that C(11) is σ-bonded to Os(2), whereas Os(3) interacts with a C(11)–C(12) π orbital. This is consistent with the C(11)–C(12) length of 1·40 (3) Å, slightly longer than the 1·33–1·34 Å expected for a C–C double bond (Bartell & Bonham, 1960).

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