O3c—Co—O2c'	96.6 (2)	Ow1—Ga—Ow2	89.3 (2)	
O3c—Co—O2c	83.4 (2)	Ow1—Ga—Ow2"	90.7 (2)	
O3c—Co—O1c	95.1 (2)	Ow1—Ga—Ow3"	90.3 (2)	
O2c—Co—O1c	84.3 (2)	Ow1—Ga—Ow3"	90.3 (2)	
O3c—Co—O1c'	84.9 (2)	Ow1—Ga—Ow3	89.7 (2)	
O2c—Co—O1c'	95.7 (2)	Ow2—Ga—Ow3	89.7 (2)	

Symmetry codes: (i) $1 - x, 1 - y, -z$; (ii) $1 - x, -y, 1 - z$.

Table 3. *Hydrogen-bonding geometry* (Å, °)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O1c—H1Oc⋯O2b'	1.031	1.954	2.984 (5)	175.8
O2c—H2Oc⋯Ow8"	0.889	1.820	2.700 (6)	169.8
O3c—H3Oc⋯O1ta'	0.807	1.920	2.723 (5)	173.5
Ow1—H1a⋯O2ta'"	0.743	1.923	2.646 (6)	164.5
Ow1—H1b⋯Ow6'"	0.817	1.850	2.661 (7)	172.1
Ow2—H2a⋯Ow5ᵛ	0.787	1.849	2.599 (7)	158.9
Ow2—H2b⋯Ow6ᵛ'	0.841	1.975	2.769 (7)	157.0
Ow3—H3a⋯O3b'"	0.830	1.795	2.594 (6)	161.2
Ow3—H3b⋯Ow7ᵛ'"	0.881	1.922	2.751 (7)	156.3
Ow4—H4a⋯O3tbⁱˣ	0.830	2.149	2.828 (6)	139.0
Ow5—H5a⋯O1b	0.801	2.022	2.801 (6)	164.3
Ow5—H5b⋯Ow4ˣ	0.848	2.089	2.811 (7)	142.8
Ow6—H6a⋯O2tbˣ'	0.841	2.217	2.932 (6)	143.0
Ow6—H6b⋯Ow4ᵛ"'	1.286	1.664	2.793 (7)	142.2
Ow7—H7a⋯O1tbᵛ"'	0.831	2.373	3.043 (6)	138.2
Ow7—H7b⋯Ow3ᵛ"'	1.113	2.215	2.751 (7)	106.8
Ow8—H8a⋯Ow7	1.043	1.916	2.866 (7)	149.8
Ow8—H8b⋯O1taˣ'	0.905	2.242	3.028 (6)	145.1

Symmetry codes: (i) $-x, 1-y, -z$; (ii) $x, y-1, z-1$; (iii) $1+x, y, 1+z$; (iv) $2 - x, 1 - y, 1 - z$; (v) $1 - x, -y, 1 - z$; (vi) $x, y - 1, z$; (vii) $1 - x, -y, -z$; (viii) $1 - x, 1 - y, 1 - z$; (ix) $x, y, 1 + z$; (x) $1 + x, y, z$; (xi) $1 + x, 1 + y, 1 + z$.

Data collection: *P4* (Siemens, 1994*a*). Cell refinement: *P4, XS-CANS* (Siemens, 1994*b*). Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990). Software used to prepare material for publication: *SHELXL93*.

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[Os5(μ-H)2(CO)16]: a Second Crystal Form

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Abstract

The title compound, hexadecacarbonyl-$1\kappa^3C,2\kappa^3C,3\kappa^3C,4\kappa^4C,5\kappa^3C$-di-$\mu$-hydrido-$1{:}2\kappa^2H;1{:}3\kappa^2H$-penta-osmium(8 *Os—Os*), has been crystallized in a different crystal form from that reported previously [Guy & Sheldrick (1978). *Acta Cryst.* B34, 1725–1727]. The form reported here has crystallographic mirror symmetry and the packing arrangement in the crystal exhibits a different relative orientation of the molecules from the earlier structure.

Comment

The title compound, (I), was first obtained by Eady, Johnson & Lewis (1973, 1977) from the vacuum pyrolysis of $Os_3(CO)_{12}$ in the presence of water. During our study of the reaction between $Os_3(CO)_{12}$ and Me_2GeH_2, we have also isolated compound (I) as a minor product. Crystallization from hot toluene gave brown, irregularly shaped crystals in the orthorhombic system.

In the earlier study by Guy & Sheldrick (1978), it was noted that the molecule has approximate C_s symmetry. In our particular sample, the mirror symmetry is crystallographically imposed, as the molecule sits on a mirror plane passing through Os1, Os3 and Os4 (Fig. 1). The molecular geometries and bond parameters are, however, otherwise identical within the e.s.d.'s reported for the earlier structure. Although the H atom was not located crystallographically, its position was calculated with the program *HYDEX* (Orpen, 1980) and it was included (but not refined) in the final cycles of refinement. The H atom was calculated to be bridging the longest Os—Os bonds (Os2—Os3 and Os2'—Os3). The closest intermolecular contacts in both the structure reported here and that reported by Guy & Sheldrick (1978) are quite similar; these involve O12 and O23 in
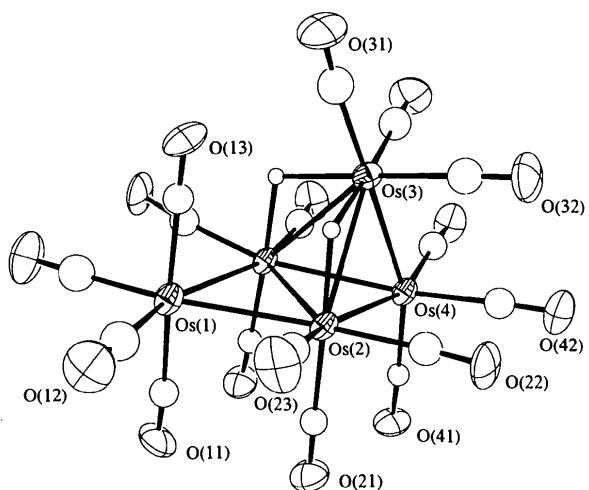
Fig. 1. *ORTEP*II (Johnson, 1976) plot of [Os₅(μ-H)₂(CO)₁₆]. Displacement ellipsoids are plotted at the 50% probability level.

our structure, and the O atoms corresponding to O12 and O22 in the earlier structure, with distances of 3.03 and 3.04 Å, respectively.

Experimental

Os₃(CO)₁₂ and excess Me₂GeH₂ were pyrolyzed in heptane at 433 K for 38 h. Chromatographic separation of the supernatant solution on silica (1:1 dichloromethane/hexane eluant) gave a small quantity of the title compound.

Crystal data
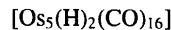
[Os₅(H)₂(CO)₁₆]	Mo $K\alpha$ radiation
$M_r = 1401.17$	$\lambda = 0.7107$ Å
Orthorhombic	Cell parameters from 25
Pmnb	reflections
$a = 10.5672\,(20)$ Å	$\theta = 15–22°$
$b = 15.9577\,(12)$ Å	$\mu = 25.96$ mm⁻¹
$c = 14.5196\,(12)$ Å	$T = 294$ K
$V = 2448.4\,(5)$ Å³	Irregular block
$Z = 4$	$0.27 \times 0.18 \times 0.14$ mm
$D_x = 3.801$ Mg m⁻³	Brown
D_m not measured	

Data collection

Enraf–Nonius CAD-4	1782 observed reflections
diffractometer	$[I_{net} > 2.5\sigma(I_{net})]$
ω/θ scans	$R_{int} = 0.078$
Absorption correction:	$\theta_{max} = 25°$
ψ scan and spherical	$h = 0 \to 12$
(North, Phillips &	$k = 0 \to 18$
Mathews, 1968)	$l = 0 \to 17$
$T_{min} = 0.0472$, $T_{max} =$	2 standard reflections
0.0884	frequency: 60 min
2975 measured reflections	intensity decay: 4%
2279 independent reflections	

Refinement

Refinement on F	$\Delta\rho_{max} = 4.7$ e Å⁻³
$R = 0.051$	$\Delta\rho_{min} = -3.5$ e Å⁻³

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\mathring{A}^2$)*

U_{iso} for C and H, $U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* a_i.a_j$ for Os and O.

	x	*y*	*z*	U_{iso}/U_{eq}
Os1	1/4	0.60168 (6)	−0.08461 (7)	0.03156
Os2	0.11701 (6)	0.75077 (3)	−0.02260 (4)	0.02318
Os3	1/4	0.76754 (6)	0.15535 (6)	0.02708
Os4	1/4	0.89823 (5)	0.02873 (6)	0.02510
O11	1/4	0.6763 (13)	−0.2792 (12)	0.0505
O12	0.0444 (16)	0.4765 (10)	−0.1410 (14)	0.0616
O13	1/4	0.5307 (11)	0.1135 (14)	0.0412
O21	0.0812 (14)	0.8188 (8)	−0.2144 (9)	0.0470
O22	−0.0975 (12)	0.8636 (9)	0.0445 (11)	0.0438
O23	−0.0943 (13)	0.6221 (9)	−0.0338 (11)	0.0453
O31	1/4	0.6250 (14)	0.2961 (16)	0.0582
O32	0.4576 (15)	0.8637 (10)	0.2536 (11)	0.0516
O41	1/4	0.9836 (12)	−0.1578 (13)	0.0415
O42	0.4478 (12)	1.0123 (8)	0.1118 (10)	0.0470
C11	1/4	0.6539 (15)	−0.2109 (18)	0.033 (5)
C12	0.1195 (20)	0.5221 (13)	−0.1213 (14)	0.049 (5)
C13	1/4	0.5554 (14)	0.0424 (16)	0.031 (5)
C21	0.0979 (15)	0.7948 (10)	−0.1452 (11)	0.027 (3)
C22	−0.0122 (18)	0.8264 (10)	0.0210 (12)	0.038 (4)
C23	−0.0105 (18)	0.6684 (11)	−0.0327 (11)	0.037 (4)
C31	1/4	0.6727 (18)	0.2433 (20)	0.045 (6)
C32	0.3799 (18)	0.8273 (11)	0.2176 (13)	0.041 (4)
C41	1/4	0.9543 (12)	−0.0873 (14)	0.021 (4)
C42	0.3751 (17)	0.9678 (10)	0.0823 (12)	0.036 (4)
H1	0.1307	0.7041	0.0936	0.0500

Table 2. *Selected geometric parameters (Å, °)*

Os1—Os2	2.906 (1)	Os3—C32	1.90 (2)
Os1—Os2ⁱ	2.906 (1)	Os4—C41	1.91 (2)
Os2—Os2ⁱ	2.811 (1)	Os4—C42	1.89 (2)
Os2—Os3	2.953 (1)	O11—C11	1.05 (3)
Os2—Os4	2.8405 (9)	O12—C12	1.11 (2)
Os3—Os4	2.780 (1)	O13—C13	1.10 (3)
Os1—C11	2.01 (3)	O21—C21	1.09 (2)
Os1—C12	1.95 (2)	O22—C22	1.13 (2)
Os1—C13	1.99 (2)	O23—C23	1.15 (2)
Os2—C21	1.92 (2)	O31—C31	1.08 (3)
Os2—C22	1.93 (2)	O32—C32	1.13 (2)
Os2—C23	1.89 (2)	O41—C41	1.13 (3)
Os3—C31	1.98 (3)	O42—C42	1.13 (2)
Os2—Os1—Os2ⁱ	57.84 (3)	Os1—Os2—C21	93.6 (5)
Os1—Os2—Os2ⁱ	61.08 (2)	Os2ⁱ—Os2—C21	96.0 (5)
Os1—Os2—Os3	96.61 (3)	Os1—C11—O11	175 (3)
Os2ⁱ—Os2—Os3	61.59 (1)	Os1—C12—O12	179 (2)
Os1—Os2—Os4	121.35 (3)	Os1—C13—O13	179 (2)
Os2ⁱ—Os2—Os4	60.35 (1)	Os2—C21—O21	177 (2)
Os3—Os2—Os4	57.31 (3)	Os2—C22—O22	172 (2)
Os2—Os3—Os2ⁱ	56.83 (3)	Os2—C23—O23	174 (2)
Os2—Os3—Os4	59.30 (3)	Os3—C31—O31	175 (3)
Os2ⁱ—Os3—Os4	59.30 (3)	Os3—C32—O32	179 (2)
Os2—Os4—Os2ⁱ	59.31 (3)	Os4—C41—O41	177 (2)
Os2—Os4—Os3	63.39 (3)	Os4—C42—O42	177 (2)
Os2ⁱ—Os4—Os3	63.39 (3)		

Symmetry code: (i) $\frac{1}{2} - x, y, z$.

The weighting scheme used , $w = w1\{1-[\Delta F/(6\Delta F_{est})]^2\}^2$, where ΔF_{est} is ΔF estimated from a Chebychev fit, is a

'robust-resistant' scheme [scheme 14 with three Chebychev coefficients in *CRYSTALS* (Watkin, Carruthers & Betteridge, 1985)]. The largest residual in the final difference map was located 0.99 Å from Os4 while largest negative residual is also near Os4, at a distance of 0.87 Å.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *NRCVAX DATRD2* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *NRCVAX SOLVER*. Program(s) used to refine structure: *CRYSTALS* (Watkin, Carruthers & Betteridge, 1985). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *NRCVAX TABLES*.

Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: BR1134). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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