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Structure of [(μ-H)Os₃(CO)₁₀(μ-COCH₂Cl)]

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Abstract. 1,1,1,1,2,2,2,3,3,3-Decacarbonyl-2,3-μ_2-chloromethoxycarbyne-2,3-μ_2-hydrido-triosmium-(3 *Os–Os*), $C_{12}H_3ClO_{11}Os_3$, $M_r = 929{\cdot}20$, monoclinic, $P2_1/c$, $a = 8{\cdot}028$ (1), $b = 17{\cdot}602$ (4), $c = 13{\cdot}805$ (3) Å, $\beta = 104{\cdot}88$ (1)°, $V = 1885{\cdot}4$ Å³, $Z = 4$, $D_x = 3{\cdot}270$ g cm⁻³, Mo $K\alpha$, $\lambda = 0{\cdot}710730$ Å, $\mu = 203{\cdot}7$ cm⁻¹, $F(000) = 1632$, room temperature, $R = 0{\cdot}021$ for 2620 reflections. The molecular structure of the title compound consists of an Os triangle, doubly bridged on one edge by a hydride and a halomethoxy ligand.

Introduction. The reaction of the osmium borylidyne carbonyl (μ-H)₃Os₃(CO)₉(μ_3-BCO) (Shore, Jan, Hsu & Hsu, 1983) with BCl₃ yields the cluster (μ-H)₃Os₃(CO)₉(μ_3-CBCl₂) (Jan, Hsu, Workman & Shore, 1987) which contains a capping —CBCl₂ unit. The Os ketenylidene (μ-H)₂Os₃(CO)₉(μ-CCO) (Sievert, Strickland, Shapley, Steinmetz & Geoffroy, 1982; Shapley, Strickland, St George, Churchill & Bueno, 1983) is both a structural and electronic (Barreto, Fehlner, Hsu, Jan & Shore, 1986) analog of the borylidyne carbonyl. It was of interest to develop the ketenylidene cluster chemistry based on the reactivity of the borylidyne carbonyl.

Here, we report the molecular structure of (μ-H)Os₃(CO)₁₀(μ-COCH₂Cl). This cluster is the result of the reaction of (μ-H)₂Os₃(CO)₉(μ_3-CCO) with BCl₃ in CH₂Cl₂.

Experimental. Light-yellow, rod-like, single crystals were obtained from CH₂Cl₂ at 258 K. For X-ray examination and data collection, a suitable crystal of approximate dimensions 0·35 × 0·25 × 0·20 mm was mounted in a glass capillary. Intensity data were collected on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation.

Unit-cell parameters were obtained by least-squares refinement of the angular settings from 25

reflections lying in a 2θ range of 24–30°. Intensity data were collected in the ω–θ scan mode with $4 \leq 2\theta \leq 50°$ under the following conditions: $0 \leq h \leq 9$, $-20 \leq k \leq 20$, $-16 \leq l \leq 16$. Six standard reflections were measured every 3 h and showed 9·4% decay. Data were corrected for Lorentz–polarization effects, intensity decay (correction factors 1·00 minimum, 1·05 maximum) and absorption (ψ scans, transmission factors 44·91% minimum, 99·86% maximum). Averaging yielded 3308 independent reflections of which 2620 with $I \geq 3\sigma(I)$ were used in the structure solution and refinement.

The structure was solved by a combination of the direct method *MULTAN*11/82 (Main *et al.*, 1982) and the difference Fourier technique, and refined by full-matrix least squares. Full-matrix least-squares refinement minimizing $w(|F_o| - |F_c|)^2$ $\{w = [\sigma(I)^2 + 0{\cdot}03(I)^2]^{-1/2}\}$ was carried out by using anisotropic thermal parameters for the non-H atoms. H(111)
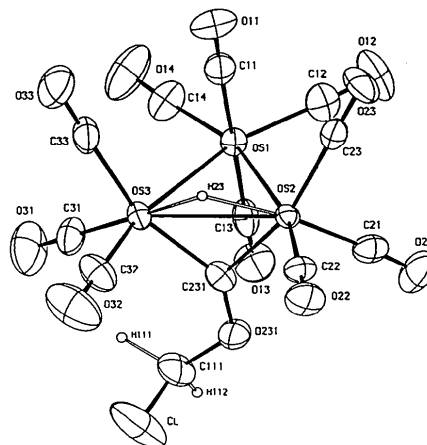

Fig. 1. *ORTEP* drawing of (μ-H)Os₃(CO)₁₀(μ-COCH₂Cl) showing 50% thermal-ellipsoid probability for the non-H atoms and atomic labelling scheme.

Table 1. *Positional parameters and B_{eq} values and their e.s.d.'s*

	x	y	z	B_{eq}^* (Å²)
Os1	0·13367 (3)	0·04738 (2)	0·81758 (2)	2·168 (5)
Os2	0·32266 (3)	0·05190 (2)	0·66863 (2)	1·840 (5)
Os3	0·15518 (3)	0·18618 (2)	0·71196 (2)	2·175 (5)
Cl	0·6238 (5)	0·2982 (2)	0·8024 (3)	8·59 (9)
O11	−0·1780 (6)	0·0155 (4)	0·6383 (4)	4·3 (1)
O12	0·1717 (9)	−0·1237 (4)	0·8543 (4)	5·5 (2)
O13	0·4568 (7)	0·0838 (4)	0·9872 (4)	4·6 (1)
O14	−0·1072 (8)	0·0972 (5)	0·9433 (4)	6·4 (2)
O21	0·6213 (7)	−0·0449 (4)	0·7843 (4)	5·1 (1)
O22	0·4800 (7)	0·0963 (4)	0·4976 (4)	3·9 (1)
O23	0·1044 (7)	−0·0814 (3)	0·5539 (4)	4·1 (1)
O31	0·1357 (9)	0·2935 (4)	0·8807 (5)	6·6 (2)
O32	0·242 (1)	0·3089 (4)	0·5793 (5)	7·3 (2)
O33	−0·2388 (8)	0·1972 (4)	0·6164 (5)	6·0 (2)
O231	0·5459 (6)	0·1567 (3)	0·8172 (4)	3·4 (1)
C11	−0·0623 (9)	0·0287 (4)	0·7040 (5)	3·0 (2)
C12	0·163 (1)	−0·0594 (5)	0·8423 (5)	3·5 (2)
C13	0·335 (1)	0·0714 (4)	0·9228 (5)	3·1 (2)
C14	−0·0134 (9)	0·0807 (5)	0·8970 (5)	3·6 (2)
C21	0·5062 (9)	−0·0114 (5)	0·7392 (5)	3·1 (2)
C22	0·4253 (8)	0·0792 (4)	0·5623 (5)	2·7 (1)
C23	0·1840 (8)	−0·0326 (4)	0·5947 (5)	2·6 (1)
C31	0·143 (1)	0·2523 (5)	0·8166 (5)	3·6 (2)
C32	0·209 (1)	0·2633 (5)	0·6291 (6)	3·9 (2)
C33	−0·094 (1)	0·1938 (5)	0·6527 (6)	3·5 (2)
C111	0·567 (1)	0·2260 (5)	0·8763 (6)	4·3 (2)
C231	0·3918 (9)	0·1418 (4)	0·7567 (4)	2·6 (1)
H23	0·17 (1)	0·123 (5)	0·628 (6)	4·0
H111	0·44 (1)	0·256 (6)	0·901 (6)	4·0
H112	0·655	0·215	0·936	5·0

* Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: $\frac{4}{3}[a^2\beta(1,1) + b^2\beta(2,2) + c^2\beta(3,3) + ab(\cos\gamma)\beta(1,2) + ac(\cos\beta)\beta(1,3) + bc(\cos\alpha)\beta(2,3)]$.

Table 2. *Selected bond distances (Å) and their e.s.d.'s for $(\mu\text{-H})Os_3(CO)_{10}(\mu\text{-COCH}_2Cl)$*

(a) Metal distances

Os(1)—Os(2)	2·855 (1)	Os(2)—Os(3)	2·858 (1)
Os(1)—Os(3)	2·873 (1)		

(b) Bridge distances

Os(2)—C(231)	1·986 (7)	C(231)—O(231)	1·330 (7)
Os(3)—C(231)	2·001 (7)	C(111)—O(231)	1·45 (1)
Os(2)—H(23)	1·76 (9)	C(111)—Cl	1·76 (1)
Os(3)—H(23)	1.63 (9)		

(c) Metal–carbon distances

Os(1)—C(11)	1·945 (6)	Os(2)—C(22)	1·921 (7)
Os(1)—C(12)	1·913 (9)	Os(2)—C(23)	1·977 (7)
Os(1)—C(13)	1·926 (6)	Os(3)—C(31)	1·877 (8)
Os(1)—C(14)	1·900 (8)	Os(3)—C(32)	1·895 (9)
Os(2)—C(21)	1·905 (7)	Os(3)—C(33)	1·963 (8)

(d) Carbonyl distances

C(11)—O(11)	1·144 (8)	C(22)—O(22)	1·133 (9)
C(12)—O(12)	1·14 (1)	C(23)—O(23)	1·131 (9)
C(13)—O(13)	1·160 (8)	C(31)—O(31)	1·16 (1)
C(14)—O(14)	1·14 (1)	C(32)—O(32)	1·13 (1)
C(21)—O(21)	1·137 (9)	C(33)—O(33)	1·14 (1)

Table 3. *Selected bond angles (°) and their e.s.d.'s for $(\mu\text{-H})Os_3(CO)_{10}(\mu\text{-COCH}_2Cl)$*

(a) Metal angles

Os(2)—Os(1)—Os(3)	59·85 (1)	Os(1)—Os(3)—Os(2)	59·76 (1)
Os(1)—Os(2)—Os(3)	60·39 (1)		

(b) Bridge angles

Os(2)—Os(1)—C(231)	40·0 (1)	C(231)—O(231)—C(111)	118·4 (6)
Os(3)—Os(1)—C(231)	40·2 (1)	O(231)—C(111)—Cl	106·9 (6)
Os(1)—Os(2)—C(231)	72·4 (2)	Os(2)—H(23)—Os(3)	115 (4)
Os(3)—Os(2)—C(231)	44·4 (2)		
Os(1)—Os(3)—C(231)	71·8 (2)		
Os(2)—Os(3)—C(231)	44·0 (2)		

(c) Metal–carbonyl angles

Os(1)—C(11)—O(11)	177·9 (7)	Os(2)—C(22)—O(22)	177·5 (6)
Os(1)—C(12)—O(12)	176·6 (7)	Os(2)—C(23)—O(23)	178·8 (6)
Os(1)—C(13)—O(13)	178·0 (7)	Os(3)—C(31)—O(31)	180 (1)
Os(1)—C(14)—O(14)	176·4 (8)	Os(3)—C(32)—O(32)	179·3 (8)
Os(2)—C(21)—O(21)	175·4 (7)	Os(3)—C(33)—O(33)	178·4 (8)

(d) Metal–metal–carbon angles

Os(2)—Os(1)—C(11)	83·9 (2)	Os(1)—Os(2)—C(23)	91·0 (2)
Os(2)—Os(1)—C(12)	95·4 (3)	Os(3)—Os(2)—C(21)	136·1 (2)
Os(2)—Os(1)—C(13)	92·1 (2)	Os(3)—Os(2)—C(22)	105·7 (2)
Os(2)—Os(1)—C(14)	158·8 (3)	Os(3)—Os(2)—C(23)	119·8 (2)
Os(3)—Os(1)—C(11)	82·8 (2)	Os(1)—Os(3)—C(31)	96·6 (3)
Os(3)—Os(1)—C(12)	154·8 (2)	Os(1)—Os(3)—C(32)	165·6 (3)
Os(3)—Os(1)—C(13)	92·7 (2)	Os(1)—Os(3)—C(33)	94·7 (2)
Os(3)—Os(1)—C(14)	99·1 (3)	Os(2)—Os(3)—C(31)	142·5 (2)
Os(1)—Os(2)—C(21)	95·7 (2)	Os(2)—Os(3)—C(32)	105·9 (3)
Os(1)—Os(2)—C(22)	166·0 (2)	Os(2)—Os(3)—C(33)	117·0 (2)

and H(23) were located directly from the difference map and refined isotropically. H(112) was calculated at C—H = 0·95 Å with a fixed isotropic thermal parameter and included in the final stages of the structure refinement. Final $R = 0·021$, $wR = 0·026$ for 2620 reflections, $S = 0·995$, $(\Delta/\sigma)_{max} = 0·35$, $(\Delta\rho)_{max} = 1·287$ e Å^{-3}. Other computer programs used: *SDP* (B. A. Frenz & Associates Inc., 1982; Enraf–Nonius, 1985). Atomic scattering factors: non-H atoms from *International Tables for X-ray Crystallography* (1974, Vol. IV), H atoms (Stewart, Davidson & Simpson, 1965). All calculations were performed on a PDP11/44 minicomputer.

Discussion. The molecular structure of $(\mu\text{-H})$-$Os_3(CO)_{10}(\mu\text{-COCH}_2Cl)$ is shown in Fig. 1. Table 1 lists the final atomic coordinates.* Selected bond distances and bond angles are collected in Tables 2 and 3, respectively. Three Os atoms defined an approximate isosceles triangle [Os(1)—Os(2) =

2·855 (1), Os(1)—Os(3) = 2·873 (1), Os(2)—Os(3) = 2·858 (1) Å] one edge of which is symmetrically bridged by the μ-halomethoxy and the μ-hydride ligand. The ten carbonyl groups are in terminal positions with four bonded to Os(1) and the remainder bonded to Os(2) and Os(3).

The basic structural unit $(\mu\text{-H})Os_3(CO)_{10}(\mu\text{-CO})$ is similar to $(\mu\text{-H})Os_3(CO)_{10}(\mu\text{-CCH}_2CHMe_2)$ (Green, Orpen & Schaverien, 1984) and $(\mu\text{-AuPPh}_3)Os_3(CO)_{10}(\mu\text{-COMe})$ (Farrugia, 1986). The ruthenium clusters $[(\mu\text{-H})Ru_3(CO)_{10}(\mu\text{-CO})]^-$

(Johnson, Lewis, Raithby & Suss, 1979), (μ-H)-Ru₃(CO)₁₀(μ-COMe) (Johnson, Lewis, Orpen, Raithby & Suss, 1979; Churchill, Beanan, Wasserman, Bueno, Rahman & Keister, 1983) and (μ-Au-PPh₃)Ru₃(CO)₁₀(μ-COMe) (Green, Mead, Mills, Salter & Stone, 1982; Bateman, Green, Mead, Mills, Slater, Stone & Woodward, 1983) and the iron cluster (μ-H)Fe₃(CO)₁₀(μ-COMe) (Shriver, Lehman & Strope, 1975) have structures similar to the Os system.

The feature of interest in (μ-H)Os₃(CO)₁₀(μ-COCH₂Cl) is the μ-halomethoxy ligand. It symmetrically spans the Os(2)—Os(3) edge along with the μ-hydride ligand. This type of multiple bridging is also observed in the clusters mentioned above. The metal—C bond distances and bond angles associated with the bridging —COCH₂Cl unit [Os(2)—C(231) = 1·986 (7), Os(3)—C(231) = 2·001 (7), C(231)—O(231) = 1·330 (7) Å,   Os(2)—C(231)—Os(3) = 91·6 (3), Os(2)—C(231)—O(231) = 127·7 (5),   Os(3)—C(231)—O(231) = 140·8 (6)°] are comparable to those found for the bridging —COMe unit found in (μ-H)Ru₃(CO)₁₀(μ-COMe) (Churchill et al., 1983) [Ru—C = 1·978–1·991,   C—O = 1·305 (5) Å, Ru—C—Ru = 90·62 (2), Ru—C—O = 128·6–140·3°] or (Johnson, Lewis, Orpen et al., 1979) [Ru—C = 1·976–1·978, C—O = 1·299 (8) Å, Ru—C—Ru = 90·3 (3),   Ru—C—O = 128·9–140·2°] and (μ-AuPPh₃Ru₃(CO)₁₀(μ-COMe) (Green et al., 1982) (Ru—C = 1·94–1·99 Å) or (μ-AuPPh₃)Os₃(CO)₁₀(μ-COMe) (Farrugia, 1986) (Os—C = 1·98–2·02, C—O = 1·28–1·30 Å, Os—C—Os = 92·0–92·2°). The title compound is also similar to the carbyne cluster (μ-H)Os₃(CO)₁₀(μ-CCH₂CHMe₂) (Green et al., 1984) with respect to the —CCH₂CHMe₂ bridging moiety [Os—C range of 1·966–2·020 Å, Os—C—Os = 92·2 (8)°]. For the Os—H—Os bridge bond distances and angles are essentially equivalent in all these clusters.

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5,5-Dichloro-10,11-dihydrodibenzo[b, f]stannepine

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Abstract. $C_{14}H_{12}Cl_2Sn$, $M_r = 369·85$, monoclinic, $P2_1/n$, $a = 11·405$ (2), $b = 8·106$ (2), $c = 16·090$ (4) Å, $\beta = 105·72$ (2)°, $V = 1431·9$ (6) Å³, $Z = 4$, $D_x = 1·716$ Mg m⁻³, $F(000) = 720$, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 2·14$ mm⁻¹, $T = 291$ (1) K, final $R = 0·023$ for 2390 unique reflexions [$F \geq 3·0\sigma(F)$].