Table 1. *Final atomic coordinates* (× 10^4, Au × 10^5) *and equivalent isotropic thermal parameters, with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{8}{3}\pi^2 \textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
Au	23649 (3)	23085 (3)	52522 (4)	3·88 (2)
P	3600 (3)	1585 (3)	7274 (3)	3·84 (12)
C(1)	1222 (10)	2946 (10)	3464 (10)	3·69 (41)
C(2)	678 (14)	3456 (11)	2514 (14)	5·40 (59)
C(3)	27 (19)	4099 (16)	1311 (16)	7·61 (84)
O(4)	212 (12)	5445 (11)	1788 (14)	8·16 (72)
C(5)	−328 (20)	6172 (16)	2812 (24)	8·63 (113)
C(11)	3146 (8)	−42 (9)	6995 (10)	3·44 (37)
C(12)	4039 (10)	−1074 (10)	7225 (12)	4·24 (47)
C(13)	3708 (12)	−2296 (11)	7070 (14)	4·89 (52)
C(14)	2500 (13)	2514 (11)	6746 (14)	4·89 (56)
C(15)	1585 (11)	−1475 (10)	6491 (14)	4·63 (50)
C(16)	1980 (11)	−257 (10)	6682 (14)	4·69 (49)
C(17)	2071 (16)	−3831 (12)	6615 (21)	7·21 (81)
C(21)	5257 (10)	1460 (10)	7668 (11)	3·66 (41)
C(22)	5590 (13)	1407 (11)	6495 (14)	5·06 (57)
C(23)	6872 (13)	1257 (12)	6760 (15)	5·06 (57)
C(24)	7775 (12)	1142 (11)	8123 (14)	4·84 (53)
C(25)	7388 (13)	1183 (13)	9268 (15)	5·55 (63)
C(26)	6139 (12)	1375 (12)	9039 (14)	4·91 (55)
C(27)	9105 (14)	897 (16)	8319 (21)	7·09 (82)
C(31)	3507 (10)	2595 (9)	9012 (13)	4·00 (45)
C(32)	3155 (9)	2130 (10)	9906 (12)	3·94 (43)
C(33)	3105 (11)	2951 (11)	11230 (14)	4·82 (53)
C(34)	3362 (12)	4283 (11)	11701 (16)	5·27 (58)
C(35)	3674 (12)	4763 (11)	10773 (14)	5·06 (55)
C(36)	3741 (12)	3928 (10)	9451 (14)	4·95 (54)
C(37)	3386 (18)	5191 (14)	13209 (17)	7·04 (81)

Table 2. *Selected bond distances* (Å) *and angles* (°)

P–Au	2·274 (1)	C(2)–C(3)	1·474 (4)
Au–C(1)	2·024 (2)	C(3)–O(4)	1·402 (4)
C(1)–C(2)	1·169 (3)	O(4)–C(5)	1·415 (4)
P–Au–C(1)	178·3 (1)	C(2)–C(3)–C(4)	113·1 (3)
Au–C(1)–C(2)	170·3 (2)	C(3)–O(4)–C(5)	113·7 (3)
C(1)–C(2)–C(3)	178·2 (3)		

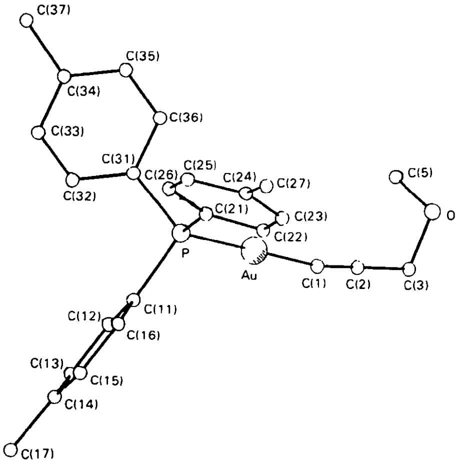

Fig. 1. One molecule of the title complex showing the atom numbering.

We thank the Spanish CAICYT for financial support and the University of Barcelona for a grant.

References

Bruce, M. I., Horn, E., Matison, J. G. & Snow, M. R. (1984). *Aust. J. Chem.* **37**, 1163–1170.

Carriedo, G. A. & Riera, V. (1988). In preparation.

Corfield, P. W. R. & Shearer, H. M. M. (1967). *Acta Cryst.* **23**, 156–162.

Cross, R. J. & Davidson, M. F. (1986). *J. Chem. Soc. Dalton Trans.* pp. 411–414.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 99–100, 149. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1988). C**44**, 979–981

Structure of a Dinuclear Osmium Complex Containing a Carboxamido and Three Bromine Ligands

By Kuang-Lieh Lu,* Ying-Chih Lin, Ming-Chu Cheng and Yu Wang

Department of Chemistry, National Taiwan University, Taipei, Taiwan

(*Received* 24 *November* 1987; *accepted* 3 *February* 1988)

Abstract. μ-Bromo-μ-(isopropylcarbamoyl-*O*,*C*)-bis(bromotricarbonylosmium), (μ-Br)$Os_2(CO)_6$[μ-OCNHCH$(CH_3)_2$]Br_2, $M_r = 874{\cdot}1$, monoclinic, $P2_1/c$, $a = 14{\cdot}579$ (3), $b = 10{\cdot}783$ (1), $c = 11{\cdot}987$ (4) Å, $\beta = 92{\cdot}08$ (2)°, $V = 1883$ (1) Å^3, $Z = 4$, $D_x = 3{\cdot}084$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 19{\cdot}831$ mm^{-1}, $F(000) = 1552$, $T = 298$ K. $R = 0{\cdot}061$ for 2358 observed reflections. The structure consists of two Os metal centers, each with three terminal carbonyl ligands, bridged by both a Br atom and a μ-η^2 carboxamido ligand; two terminal Br atoms are *trans* to each other. The two Os atoms are separated by 3·956 (2) Å, indicating no metal–metal interaction.

---

* Permanent address: Institute of Chemistry, Academia Sinica, Nankang, Taipei, Taiwan.

0108-2701/88/060979-03$03.00

Introduction. The chemistry of the hydrido carboxamido triosmium cluster $(\mu\text{-H})Os_3(CO)_{10}[\mu\text{-OCNHCH}(CH_3)_2]$ (1) has been little explored, probably owing to its high thermal stability. Since halide has been employed as a promoter in many catalytic (Dombek, 1981) or stoichiometric reactions (Morrison, Geoffroy & Rheingold, 1985), we were interested in the chemical behavior of the cluster in the presence of halogen. In this work, the crystal structure of the product, $(\mu\text{-Br})Os_2(CO)_6[\mu\text{-OCNHCH}(CH_3)_2]Br_2$, from the reaction of (1) with Br_2 has been determined.

Experimental. Reaction of $(\mu\text{-H})Os_3(CO)_{10}[\mu\text{-OCNHCH}(CH_3)_2]$ with Br_2 in chloroform solution at room temperature gives a white precipitate. This complex transforms to a yellow compound in chloroform solution over a one week period. Crystals of the yellow complex were grown by diffusing *n*-hexane vapor into a saturated dichloromethane solution of the complex at room temperature. Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$. Crystal 0·10 × 0·15 × 0·25 mm. Unit-cell parameters from 25 reflections with $18{\cdot}6 < 2\theta < 21{\cdot}1°$, data collected by $\omega/2\theta$ scan method with scan width in 2θ of $2(0{\cdot}7 + 0{\cdot}35\tan\theta)°$ and variable scan speed of 20/3–20/23° min^{-1}, variation of standard reflections checked every 2 h, <4%. $2\theta_{\max} = 50°$ $(0 \le h \le 14,$ $0 \le k \le 12,$ $-17 \le l \le 17)$, 3684 unique reflections, 2358 observed with $I > 2{\cdot}5\sigma(I)$. Empirical absorption correction based on azimuthal rotation from three reflections (North, Phillips & Mathews, 1968); transmission factors 0·17–1·00. Structure solved by heavy-atom method. The function minimized in least-squares refinement was $\sum w(|F_o| - |F_c|)^2$, where $w = 1/\sigma^2(F_o)$, $\sigma(F_o)$ from counting statistics. 209 parameters with anisotropic temperature factors for non-H atoms. $R = 0{\cdot}061$, $wR = 0{\cdot}048$, $S = 2{\cdot}8827$, $(\Delta/\sigma)_{\max} = 0{\cdot}03$ in the final cycle. $\Delta\rho_{\max} = 3{\cdot}1$ e Å^{-3} near Os. Atomic scattering factors and anomalous-dispersion terms from *International Tables for X-ray Crystallography* (1974).

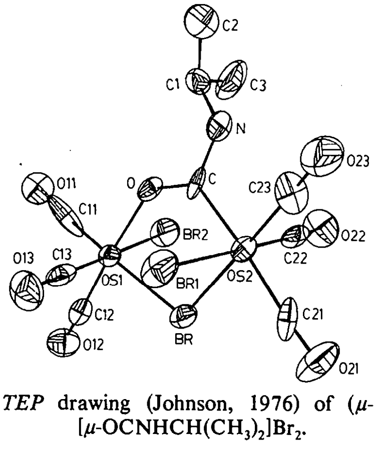

Fig. 1. *ORTEP* drawing (Johnson, 1976) of $(\mu\text{-Br})Os_2(CO)_6[\mu\text{-OCNHCH}(CH_3)_2]Br_2$.

Table 1. *Atomic coordinates and equivalent isotropic temperature factors* (Å^2)

$$B_{eq} = \tfrac{8}{3}\pi^2 \textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Os1	0·63633 (7)	0·23190 (7)	0·25471 (8)	2·69 (4)
Os2	0·89150 (7)	0·35395 (7)	0·24034 (8)	2·70 (4)
Br	0·77223 (18)	0·22753 (20)	0·12555 (20)	3·48 (10)
Br1	0·91405 (21)	0·16869 (22)	0·37101 (22)	4·49 (12)
Br2	0·57228 (22)	0·41365 (22)	0·14113 (24)	4·88 (14)
C	0·7830 (13)	0·4138 (16)	0·3364 (17)	2·2 (9)
O	0·7065 (10)	0·3640 (11)	0·3489 (11)	2·7 (7)
N	0·7901 (13)	0·5236 (14)	0·3963 (16)	3·5 (9)
C1	0·7171 (17)	0·5898 (17)	0·4532 (20)	3·9 (12)
C2	0·7559 (17)	0·6346 (21)	0·5649 (24)	5·2 (14)
C3	0·6881 (24)	0·6904 (22)	0·380 (3)	7·2 (19)
C11	0·5400 (16)	0·2453 (19)	0·338 (3)	6·3 (16)
O11	0·4737 (12)	0·2579 (14)	0·3965 (15)	5·4 (10)
C12	0·5707 (15)	0·1212 (18)	0·1585 (18)	3·1 (10)
O12	0·5310 (14)	0·0549 (14)	0·1032 (14)	5·7 (10)
C13	0·6852 (17)	0·0972 (18)	0·3425 (17)	3·3 (11)
O13	0·7087 (15)	0·0116 (13)	0·3901 (15)	5·9 (11)
C21	0·9909 (16)	0·2870 (18)	0·1536 (20)	3·7 (11)
O21	1·0558 (13)	0·2495 (16)	0·1095 (10)	5·8 (10)
C22	0·8674 (15)	0·4914 (17)	0·1412 (18)	2·9 (10)
O22	0·8520 (14)	0·5706 (13)	0·0876 (15)	5·7 (10)
C23	0·9769 (16)	0·4391 (18)	0·3279 (22)	4·0 (12)
O23	1·0311 (14)	0·4852 (15)	0·3844 (15)	6·1 (11)

Computing programs: *NRCC SDP* PDP-11 package (Gabe & Lee, 1981).

Discussion. The molecular structure of the complex is shown in Fig. 1. The atomic coordinates and B_{eq}'s are listed in Table 1.* Selected interatomic distances and angles are given in Table 2. In this complex, two Os metal centers, each with three terminal carbonyl ligands and one terminal Br atom, are bridged by both a Br atom and a μ-η^2 carboxamido ligand, With two Os metal atoms, the central part of the structure presents a five-membered ring, and the two terminal Br atoms are *trans* to each other. A pseudo-octahedral geometry was observed around each Os. The two Os atoms are separated by 3·956 (2) Å, indicating no metal–metal interaction. When two metals are bridged by the carboxamido and a hydride ligand, where a metal–metal bond is assumed, such as that in $HOs_3(CO)_{10}$-(OCNHR), the metal–metal separation was found to be in the range of 2·6–3·0 Å (Lin, Mayr, Knobler & Kaesz, 1984). The μ-η^2 carboxamido ligand in (1) exhibits higher stability towards the Br reaction than the $Os(CO)_4$ group which is also bridging the same two Os atoms with the fairly strong Os—Os bonds. Such stability could be attributed to the soft-acid–soft-base interaction. Halogen atoms can serve as a bridge ligand between two non-bonded metal atoms, and an *M*—*X*—*M* angle of greater than 100° has been reported

---

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44747 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Selected bond lengths* (Å) *and bond angles* (°)

Os1–Br	2·559 (2)	Os1–Br2	2·544 (2)
Os1–O	2·07 (1)	OS1–C11	1·76 (3)
Os1–C12	1·89 (2)	Os1–C13	1·92 (2)
Os2–Br	2·569 (2)	Os2–Br1	2·552 (2)
Os2–C	2·09 (1)	Os2–C21	1·95 (2)
Os2–C22	1·92 (2)	Os2–C23	1·84 (2)
C–O	1·25 (2)	C–N	1·39 (2)
N–Cl	1·47 (2)	Cl–C2	1·51 (3)
C1–C3	1·45 (3)	C11–O11	1·22 (3)
C12–O12	1·12 (2)	C13–O13	1·13 (2)
C21–O21	1·17 (2)	C22–O22	1·09 (2)
C23–O23	1·14 (3)		

Br–Os1–Br2	88·1 (1)	Br–Os1–O	88·0 (3)
Br–Os1–C11	175·6 (7)	Br–Os1–C12	90·3 (6)
Br–Os1–C13	92·2 (6)	Br2–Os1–O	85·8 (3)
Br2–Os1–C11	87·4 (7)	Br2–Os1–C12	89·6 (6)
Br2–Os1–C13	178·9 (5)	O–Os1–C11	91·3 (8)
O–Os1–C12	175·1 (7)	O–Os1–C13	93·2 (7)
C11–Os1–C12	90·(1)	C11–Os1–C13	92·(1)
C12–Os1–C13	91·5 (8)	Br–Os2–Br1	89·0 (1)
Br–Os2–C	87·0 (5)	Br–Os2–C21	91·1 (7)
Br–Os2–C22	88·4 (6)	Br–Os2–C23	177·3 (6)
Br1–Os2–C	89·2 (4)	Br1–Os2–C21	87·5 (5)
Br1–Os2–C22	176·8 (6)	Br1–Os2–C23	88·3 (6)
C–Os2–C21	176·2 (7)	C–Os–C22	88·7 (7)
C–Os2–C23	92·4 (9)	C21–Os2–C22	94·5 (8)
C21–Os2–C23	89· (1)	C22–Os2–C23	94·2 (9)
Os1–Br–Os2	101·0 (1)	Os2–C–O	129· (1)
Os2–C–N	120· (1)	O–C–N	111· (1)
Os1–O–C	131· (1)	C–N–C1	128· (1)
N–C1–C2	108· (2)	N–C1–C3	106· (1)
C2–C1C3	113· (1)	Os1–C11–O11	178· (1)
OS1–C12–O12	179· (1)	Os1–C13–O13	174· (2)
Os2–C21–O21	174· (2)	Os2–C22–O22	178· (1)
Os2–C23–O23	176· (1)		

(Morrison, Geoffroy, Rheingold & Fultz, 1985). In our compound the Os1–Br–Os2 angle is 101·0(1)°. The Os–C(CO) distances are somewhat shorter for two CO ligands *trans* to the bridged Br ligand [Os2–C23, 1·84 (2), Os1–C11, 1·76 (3) Å]. Similar phenomena were observed previously (Churchill & Lashewycz, 1979). Since both Os metal atoms are in a formally divalent oxidation state, the corresponding C–O distances are normal, and the relatively shorter Os–C distance may be due to the stronger σ donor ability of the CO ligand affected by the unique *trans* influence of the bridged Br ligand. The carboxamido ligand shows partial double-bond character for both C=O [1·25 (2) Å] and CN [1·39 (2) Å] bonds, similar to many other complexes of the same type (Szostak, Strouse & Kaesz, 1980). The hindered rotation about the C–N bond was indicated by the nonequivalence of the two methyl groups in the NMR spectrum. The carboxamido group (OCN) is planar with the O–C–N angle smaller than for a normal sp^2 hybrid.

This work was supported by the National Science Council, Taiwan.

References

Churchill, M. R. & Lashewycz, R. A. (1979). *Inorg. Chem.* **18**, 1926–1930.

Dombek, B. D. (1981). *J. Am. Chem. Soc.* **103**, 6508–6510.

Gabe, E. J. & Lee, F. L. (1981). *Acta Cryst.* A**37**, C339.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Lin, Y. C., Mayr, A., Knobler, C. B. & Kaesz, H. D. (1984). *J. Organomet. Chem.* **272**, 207–229.

Morrison, E. D., Geoffroy, G. L. & Rheingold, A. L. (1985). *J. Am. Chem. Soc.* **107**, 254–255.

Morrison, E. D., Geoffroy, G. L., Rheingold, A. L. & Fultz, W. C. (1985). *Organometallics*, **4**, 1413–1418.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Szostak, R., Strouse, C. E. & Kaesz, H. D. (1980). *J. Organomet. Chem.* **191**, 243–260.

Acta Cryst. (1988). C**44**, 981–986

Polydentate Chelates. 3. Structures of egta^{4-} Chelates of Manganese(II) and Copper(II): Sr[Mn(egta)].7H$_2$O and [Cu$_2$(egta)(OH$_2$)$_2$].2H$_2$O

By Cynthia K. Schauer and Oren P. Anderson*

Department of Chemistry, Colorado State University, Fort Collins, Colorado 80523, *USA*

(*Received* 5 *November* 1987; *accepted* 3 *February* 1988)

Abstract. Compound (1): Sr[Mn($C_{14}H_{20}N_2O_{10}$)].-7H_2O, $M_r = 645{\cdot}0$, monoclinic, $P2_1/n$, $a = 16{\cdot}535$ (4), $b = 8{\cdot}415$ (2), $c = 17{\cdot}457$ (4) Å, $\beta = 97{\cdot}11$ (2)°, $V = 2410{\cdot}3$ (5) Å^3, $Z = 4$, $D_x = 1{\cdot}78$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 28{\cdot}97$ cm^{-1}, $F(000) = 1324$, $T = 143$ K. Compound (2): [$Cu_2(C_{14}H_{20}N_2O_{10})(H_2O)_2$].2$H_2O$, $M_r = 575{\cdot}5$, monoclinic, $C2/c$, $a = 20{\cdot}962$ (5), $b = 7{\cdot}513$ (2), $c = 13{\cdot}545$ (2) Å, $\beta = 90{\cdot}85$ (2)°, $V = 2132{\cdot}9$ (4) Å^3, $Z = 4$, $D_x = 1{\cdot}79$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 20{\cdot}71$ cm^{-1}, $F(000) = 1184$, $T = 143$ K. For (1), $R = 0{\cdot}036$ for 3421 unique observed reflections; for (2), $R = 0{\cdot}031$ for 1670 unique observed reflections.

---

* To whom all correspondence should be addressed.

0108-2701/88/060981-06$03.00