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

O–U	2·096 (6)	C6–U	2·806 (9)
O′–U	2·129 (5)	C9–U	2·736 (9)
Cp1–U	2·499	C7–U	2·795 (9)
Cp2–U	2·494	C8–U	2·729 (9)
C1–U	2·735 (9)	C10–U	2·793 (9)
C2–U	2·778 (8)	U–U′	3·3927 (9)
C3–U	2·784 (9)	O–O′	2·520 (11)
C4–U	2·781 (9)	⟨Si–C⟩	1·857 (21)
C5–U	2·796 (9)		

Cp1–U–Cp2	123·38	O′–U–Cp2	109·40
O–U–Cp1	115·11	O–U–O	73·20 (23)
O–U–Cp2	109·78	U–O–U	106·80 (23)
O′–U–Cp1	115·14		

Cp1 and Cp2 are the centroids of atoms C(1)–C(5), C(6)–C(10) respectively. Primed atoms are at −*x*, −*y*, −*z*.

International Tables for X-ray Crystallography (1974); local unpublished programs and *ORTEP* (Johnson, 1965).

Atomic parameters are listed in Table 1,* and distances and angles are listed in Table 2. Fig. 1 shows the molecule and the numbering scheme.

Related literature. The geometry reported here is in agreement with that of an independent, but sparsely reported, determination (Blake, Lappert, Taylor, Attwood & Zhang, 1987). The Si atoms are from 0·3 to 0·5 Å out of the plane of the cyclopentadienyl rings and away from the U atoms.

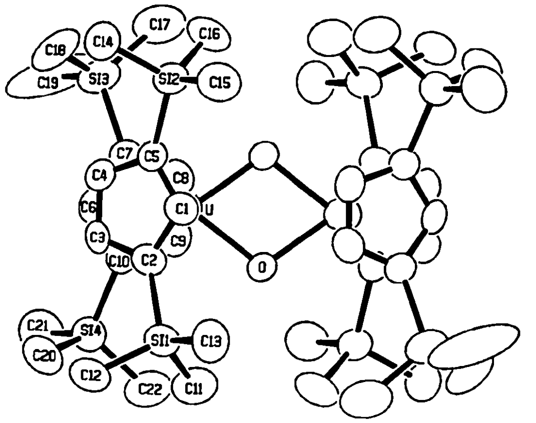

Fig. 1. *ORTEP* drawing of title complex, with thermal ellipsoids at 50% probability level.

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* Lists of structure factors, anisotropic thermal parameters, calculated hydrogen positions, distances and angles, and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51071 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Acetatobromocarbonylbis(triphenylphosphine)osmium(II)

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Abstract. $[OsBr(CO)(CH_3CO_2)\{P(C_6H_5)_3\}_2]$, $M_r = 881·74$, monoclinic $P2_1/n$, $a = 9·751\ (3)$, $b = 20·334\ (4)$, $c = 17·458\ (2)$ Å, $\beta = 96·42\ (1)°$, $V = 3440\ (1)$ Å^3, $Z = 4$, $D_x = 1·70$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0·71069$ Å, $\mu = 53·32$ cm^{-1}, $F(000) = 1728$, $T = 295$ K, $R = 0·046$, 3293 unique observed reflections. The compound was prepared by refluxing di(tetra-*n*-butylammonium) hexabromoosmate(IV) and triphenylphosphine in mixed acetic acid/acetic anhydride. The central Os atom is in distorted octahedral coordi-

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Table 1. *Positional and equivalent isotropic thermal parameters for the non-*H *atoms and their e.s.d.'s*

$$B_{eq} = (8\pi^2/3)[U_{22} + (1/\sin^2\beta)(U_{11} + U_{33} + 2U_{13}\cos\beta)].$$

	x	y	z	B_{eq}(Å^2)
Os(1)	0·94891 (6)	0·21602 (3)	0·15256 (3)	1·86 (2)
Br(1)	1·0782 (2)	0·10725 (9)	0·1757 (1)	4·00 (8)
P(1)	0·8692 (4)	0·3249 (2)	0·1531 (2)	2·1 (2)
P(2)	0·7429 (4)	0·1561 (2)	0·1388 (2)	2·2 (2)
O(1)	1·013 (1)	0·2315 (4)	0·2767 (5)	2·5 (4)
O(2)	1·153 (1)	0·2523 (5)	0·1902 (7)	2·9 (5)
O(3)	0·959 (1)	0·2225 (6)	−0·0166 (6)	4·0 (6)
C(1)	0·805 (1)	0·3646 (8)	0·0632 (9)	2·8 (7)
C(2)	0·839 (2)	0·4295 (8)	0·047 (1)	5 (1)
C(3)	0·783 (2)	0·459 (1)	−0·020 (1)	6 (1)
C(4)	0·696 (2)	0·426 (1)	−0·074 (1)	5 (1)
C(5)	0·657 (2)	0·364 (1)	−0·057 (1)	5 (1)
C(6)	0·719 (2)	0·3330 (7)	0·009 (1)	3·3 (7)
C(7)	0·739 (1)	0·3452 (8)	0·2169 (9)	2·8 (7)
C(8)	0·715 (2)	0·3053 (8)	0·276 (1)	3·1 (7)
C(9)	0·624 (2)	0·324 (1)	0·329 (1)	5 (1)
C(10)	0·565 (2)	0·383 (1)	0·324 (1)	5 (1)
C(11)	0·592 (2)	0·426 (1)	0·266 (1)	5 (1)
C(12)	0·677 (2)	0·4061 (7)	0·212 (1)	3·5 (8)
C(13)	1·011 (1)	0·3795 (7)	0·192 (1)	2·6 (7)
C(14)	1·122 (2)	0·3868 (8)	0·149 (1)	3·3 (8)
C(15)	1·236 (2)	0·4273 (9)	0·178 (1)	4·3 (9)
C(16)	1·234 (2)	0·4569 (9)	0·249 (1)	5 (1)
C(17)	1·124 (2)	0·449 (1)	0·289 (1)	5 (1)
C(18)	1·015 (2)	0·4118 (8)	0·261 (1)	3·6 (8)
C(19)	0·749 (1)	0·0782 (6)	0·0878 (8)	1·8 (6)
C(20)	0·658 (2)	0·025 (1)	0·103 (1)	5 (1)
C(21)	0·653 (3)	−0·028 (1)	0·060 (1)	6 (1)
C(22)	0·732 (2)	−0·0394 (8)	0·002 (1)	4 (1)
C(23)	0·821 (2)	0·009 (1)	−0·014 (1)	5 (1)
C(24)	0·828 (2)	0·066 (1)	0·029 (1)	4·2 (9)
C(25)	0·589 (1)	0·1926 (7)	0·085 (1)	2·7 (7)
C(26)	0·558 (2)	0·1784 (9)	0·007 (1)	3·5 (8)
C(27)	0·449 (2)	0·2084 (8)	−0·038 (1)	4·0 (8)
C(28)	0·370 (2)	0·251 (1)	−0·003 (1)	5 (1)
C(29)	0·395 (2)	0·266 (1)	0·073 (1)	5 (1)
C(30)	0·506 (2)	0·2378 (7)	0·118 (1)	2·8 (7)
C(31)	0·683 (2)	0·1329 (8)	0·231 (1)	3·1 (7)
C(32)	0·544 (2)	0·1259 (9)	0·241 (1)	4 (1)
C(33)	0·507 (2)	0·108 (1)	0·313 (1)	5 (1)
C(34)	0·604 (3)	0·100 (1)	0·374 (1)	6 (1)
C(35)	0·741 (2)	0·103 (1)	0·365 (1)	5 (1)
C(36)	0·780 (2)	0·1204 (9)	0·294 (1)	4·0 (9)
C(37)	0·952 (1)	0·2187 (8)	0·0476 (8)	2·7 (6)
C(38)	1·134 (2)	0·2485 (7)	0·261 (1)	3·0 (7)
C(39)	1·246 (2)	0·269 (1)	0·321 (1)	5 (1)

Table 2. *Selected bond distances* (Å), *bond angles* (°), *and their e.s.d.'s*

Os(1)—C(37)	1·84 (1)	P(1)—C(13)	1·84 (1)
Os(1)—O(2)	2·15 (1)	P(2)—C(19)	1·82 (1)
Os(1)—O(1)	2·21 (1)	P(2)—C(31)	1·83 (2)
Os(1)—P(2)	2·339 (4)	P(2)—C(25)	1·84 (2)
Os(1)—P(1)	2·347 (4)	O(1)—C(38)	1·29 (2)
Os(1)—Br(1)	2·555 (2)	O(2)—C(38)	1·27 (2)
P(1)—C(1)	1·81 (2)	O(3)—C(37)	1·13 (2)
P(1)—C(7)	1·83 (1)		

C(37)—Os(1)—O(2)	100·3 (5)	C(7)—P(1)—C(13)	100·8 (7)
C(37)—Os(1)—O(1)	160·0 (5)	C(19)—P(2)—C(31)	103·7 (7)
C(37)—Os(1)—P(2)	91·2 (5)	C(19)—P(2)—C(25)	100·0 (7)
C(37)—Os(1)—P(1)	91·0 (5)	C(31)—P(2)—C(25)	103·4 (8)
C(37)—Os(1)—Br(1)	97·0 (5)	C(1)—P(1)—Os(1)	119·9 (6)
O(2)—Os(1)—O(1)	60·0 (4)	C(7)—P(1)—Os(1)	118·0 (5)
O(2)—Os(1)—P(2)	164·4 (3)	C(13)—P(1)—Os(1)	109·5 (5)
O(2)—Os(1)—P(1)	88·3 (3)	C(19)—P(2)—Os(1)	115·2 (5)
O(2)—Os(1)—Br(1)	80·0 (3)	C(25)—P(2)—Os(1)	119·0 (5)
O(1)—Os(1)—P(2)	108·8 (3)	C(31)—P(2)—Os(1)	113·5 (6)
O(1)—Os(1)—P(1)	85·4 (3)	C(38)—O(1)—Os(1)	89 (1)
O(1)—Os(1)—Br(1)	83·5 (2)	C(38)—O(2)—Os(1)	93 (1)
P(2)—Os(1)—P(1)	102·2 (1)	O(3)—C(37)—Os(1)	176 (1)
P(2)—Os(1)—Br(1)	88·2 (1)	O(2)—C(38)—O(1)	118 (1)
P(1)—Os(1)—Br(1)	166·8 (1)	O(2)—C(38)—C(39)	120 (2)
C(1)—P(1)—C(7)	103·5 (7)	O(1)—C(38)—C(39)	122 (2)
C(1)—P(1)—C(13)	102·6 (7)		

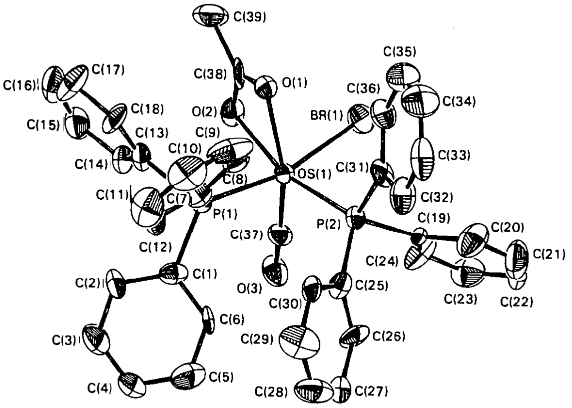

Fig. 1. Molecular structure and numbering scheme. Thermal ellipsoids at 50% probability level, H atoms omitted for clarity.

nation with the Br atom, the CO ligand, the two O atoms of the chelated acetate ligand, and the two P atoms of the triphenylphosphine groups.

Experimental. Preparation described by Robinson, Hinckley & Ikuo (1988). This compound is a minor component of the earlier preparation. Crystals obtained by layering hexane over solution. Nearly colorless light brown crystals found among more abundant brown crystals. Crystal used for data collection 0·15 × 0·13 × 0·09 mm, broken fragment, nearly colorless, slight brownish tint. Rigaku AFC5S diffractometer, graphite-monochromated Mo $K\alpha$ radiation, ω–2θ scans, scan speed 8° min^{-1}, maximum of three scan repetitions to obtain $\sigma(F)/F < 0\cdot10$. Lattice parameters from least-squares fit of 25 strong reflections in 2θ range 20–29°. 6645 reflections measured (h 0 to 11, k 0 to 24, l −20 to 20), 583 redundant and/or systematically extinct reflections deleted, data set comprised of 6062 unique reflections. 3293 reflections were considered observed [$I > 3\sigma(I)$], $R_{int} = 2\cdot8\%$ for 387 equivalent reflections, $(\sin\theta)/\lambda_{max} = 0\cdot60$ Å^{-1}. Three standard reflections ($0\bar{1}\bar{2}$, $\bar{1}0\bar{1}$, $00\bar{2}$) varied by −1·0, −0·4 and −0·1%, respectively; no decay correction applied. Data corrected for Lorentz, polarization and absorption (empirical ψ-scan method, five reflections, transmission range 0·84–1·0). Direct methods provided the locations of Os, P and most of the ring C atoms, remaining non-H atoms found by least-squares/Fourier methods. Full-matrix least-squares refinement of F magnitudes performed on 415 variables including all non-H positional and anisotropic thermal parameters and one scale factor. Pyridine-ring and methyl H atoms placed in geometrically correct positions (C—H = 0·95 Å) but not refined; one methyl H atom, found by difference Fourier synthesis, was used to fix the orientation of the CH_3 group. H-atom B's fixed at 1·2 × B_{eq} of associated C atom. Convergence yielded $R = 0\cdot046$, $wR = 0\cdot068$

$[w = 1/\sigma^2(|F_o|)]$, $S = 1\cdot24$, and $(\Delta/\sigma)_{\max} = 0\cdot37$. A final difference Fourier synthesis showed $(\Delta\rho)_{\max} = 1\cdot13$ and $(\Delta\rho)_{\min} = -0\cdot86$ e Å^{-3}, both in the vicinity of the Os atom. Atomic scattering factors and anomalous-dispersion corrections from Cromer & Waber (1974). Atomic coordinates and temperature factors given in Table 1, selected interatomic distances and angles presented in Table 2. Fig. 1 shows the molecular configuration, thermal vibrations and the atom-numbering scheme.* All computer programs from the *TEXSAN* crystal structure analysis package (Molecular Structure Corporation, 1985).

Related literature. The compound is one of a series of related compounds obtained through the reduction of the hexabromoosmate(IV) ion by triphenylphosphine (Robinson, Hinckley & Ikuo, 1988; Hinckley, Matusz, Kibala & Robinson, 1987).

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* Lists of structure factors, anisotropic thermal parameters, distances and angles involving the phenyl rings, H-atom coordinates, torsion angles, intermolecular distances, and selected least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51121 (48 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1988). C**44**, 1829–1831

Structure of *mer*-Tribromotris(triphenylstibine)osmium(III)

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(*Received* 9 *May* 1988; *accepted* 9 *June* 1988)

Abstract. $\{OsBr_3[Sb(C_6H_5)_3]_3\}$, $M_r = 1489\cdot11$, monoclinic, $P2_1/n$, $a = 18\cdot713\,(4)$, $b = 13\cdot204\,(3)$, $c = 19\cdot987\,(7)$ Å, $\beta = 91\cdot56\,(3)°$, $V = 4937\,(4)$ Å^3, $Z = 4$, $D_x = 2\cdot00$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 69\cdot70$ cm^{-1}, $F(000) = 2812$, $T = 295$ K, $R = 0\cdot055$, 4010 unique observed reflections. Synthesis by reaction of hexabromoosmate(IV) ion with triphenylstibine in mixed solvent acetic acid/acetic anhydride in the presence of acetate. The Os is six coordinate, bound to three Br ligands and three Sb atoms of the triphenylstibine ligands in a *mer* configuration. The three Os—Sb distances are 2·640 (2), 2·644 (2) and 2·654 (2) Å.

Experimental. Prepared by combining di(tetra-*n*-butylammonium) hexabromoosmate(IV) (0·5 g), sodium acetate trihydrate (0·5 g), and triphenylstibine (0·5 g) in a mixed solvent of 5 ml acetic acid and 5 ml acetic anhydride. Mixture refluxed 10 min and filtered while hot. Black solid product washed with acetic acid. Crystals from methylene chloride solution layered with hexane. Crystal used for data collection 0·19 × 0·15 × 0·13 mm fragment cut from larger black crystal. Rigaku AFC5S diffractometer, graphite-monochromated Mo $K\alpha$ radiation, ω–2θ scans, scan speed 6° min^{-1}, maximum three scan repetitions to obtain $\sigma(F)/F < 0\cdot10$. Lattice parameters from least-squares fit of 21 reflections in 2θ range 6–23°. 9087 reflections measured (h −22 to 22, k 0 to 15, l 0 to 23), 420 redundant and/or systematically extinct reflections deleted, data set comprised of 8667 unique reflections. 4010 reflections were considered observed $[I > 3\sigma(I)]$, $R_{\rm int} = 0\cdot9\%$ for 279 equivalent reflections, $(\sin\theta)/\lambda_{\max} = 0\cdot60$ Å^{-1}. Three standard reflections ($\bar{2}11$, $\bar{2}\bar{1}\bar{1}$, $\bar{4}02$) varied by −9·13, −9·19, and −8·44% respectively; decay correction applied. Data corrected for Lorentz, polarization and absorption (empirical ψ-scan method, five reflections, transmission range 0·82–1·0).

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