$[w = 1/\sigma^2(|F_o|)]$, $S = 1{\cdot}24$, and $(\Delta/\sigma)_{\max} = 0{\cdot}37$. A final difference Fourier synthesis showed $(\Delta\rho)_{\max} = 1{\cdot}13$ and $(\Delta\rho)_{\min} = -0{\cdot}86$ 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).

This work was supported by the Southern Illinois University Materials Technology Center.

References

CROMER, D. T. & WABER, J. T. (1974). In *International Tables for X-ray Crystallography*, Vol. IV, p. 71 (scattering factors), p. 148 (anomalous dispersion). Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

HINCKLEY, C. C., MATUSZ, M., KIBALA, P. A. & ROBINSON, P. D. (1987). *Acta Cryst.* C**43**, 1880–1882.

Molecular Structure Corporation (1985). *TEXSAN. Structure Analysis Package.* MSC, 3304 Longmire Drive, College Station, TX 77840, USA.

ROBINSON, P. D., HINCKLEY, C. C. & IKUO, A. (1988). *Acta Cryst.* C**44**, 1491–1492.

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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.

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

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Abstract. $\{OsBr_3[Sb(C_6H_5)_3]_3\}$, $M_r = 1489{\cdot}11$, monoclinic, $P2_1/n$, $a = 18{\cdot}713\ (4)$, $b = 13{\cdot}204\ (3)$, $c = 19{\cdot}987\ (7)$ Å, $\beta = 91{\cdot}56\ (3)^\circ$, $V = 4937\ (4)$ Å^3, $Z = 4$, $D_x = 2{\cdot}00$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 69{\cdot}70$ cm^{-1}, $F(000) = 2812$, $T = 295$ K, $R = 0{\cdot}055$, 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 $\sigma(F)/F < 0{\cdot}10$. 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 $[I > 3\sigma(I)]$, $R_{\rm int} = 0{\cdot}9\%$ for 279 equivalent reflections, $(\sin\theta)/\lambda_{\max} = 0{\cdot}60$ Å^{-1}. Three standard reflections ($\bar{2}11$, $\overline{211}$, $\bar{4}02$) 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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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	0·77299 (4)	0·06912 (6)	0·04290 (4)	1·99 (3)
Sb(1)	0·75767 (7)	0·1198 (1)	−0·08467 (6)	2·27 (6)
Sb(2)	0·75944 (7)	−0·1274 (1)	0·01790 (6)	2·31 (6)
Sb(3)	0·80372 (7)	0·2541 (1)	0·08473 (6)	2·43 (6)
Br(1)	0·6435 (1)	0·1171 (2)	0·0465 (1)	3·5 (1)
Br(2)	0·9049 (1)	0·0364 (2)	0·0399 (1)	3·2 (1)
Br(3)	0·7645 (1)	0·0174 (2)	0·1639 (1)	3·7 (1)
C(1)	0·6782 (6)	0·0362 (9)	−0·1422 (6)	2·5 (4)
C(2)	0·6844 (6)	0·0281 (10)	−0·2114 (6)	4·3 (5)
C(3)	0·6308 (8)	−0·0197 (11)	−0·2496 (5)	5·2 (6)
C(4)	0·5710 (7)	−0·0594 (11)	−0·2186 (7)	5·3 (6)
C(5)	0·5649 (6)	−0·0514 (10)	−0·1494 (7)	5·0 (6)
C(6)	0·6185 (7)	−0·0036 (10)	−0·1112 (5)	3·1 (4)
C(7)	0·7132 (6)	0·2644 (8)	−0·1071 (6)	2·1 (4)
C(8)	0·6390 (6)	0·2728 (8)	−0·1132 (7)	3·6 (5)
C(9)	0·6077 (5)	0·3662 (11)	−0·1282 (7)	4·5 (5)
C(10)	0·6506 (7)	0·4513 (8)	−0·1371 (7)	5·0 (6)
C(11)	0·7248 (7)	0·4430 (8)	−0·1311 (7)	4·4 (5)
C(12)	0·7561 (5)	0·3495 (1)	−0·1161 (7)	3·7 (5)
C(13)	0·8444 (6)	0·1184 (10)	−0·1514 (5)	2·3 (4)
C(14)	0·8422 (6)	0·1826 (9)	−0·2068 (6)	3·3 (4)
C(15)	0·8974 (7)	0·1806 (10)	−0·2523 (5)	3·9 (5)
C(16)	0·9548 (6)	0·1144 (11)	−0·2424 (6)	4·2 (5)
C(17)	0·9570 (6)	0·0502 (9)	−0·1869 (7)	4·6 (5)
C(18)	0·9018 (7)	0·0522 (9)	−0·1414 (5)	2·8 (4)
C(19)	0·6565 (5)	−0·1948 (10)	0·0024 (6)	2·3 (4)
C(20)	0·6440 (6)	−0·2781 (10)	−0·0392 (6)	3·6 (5)
C(21)	0·5747 (7)	−0·3151 (9)	−0·0491 (6)	4·2 (5)
C(22)	0·5178 (5)	−0·2687 (11)	−0·0173 (7)	4·5 (5)
C(23)	0·5303 (6)	−0·1854 (10)	0·0243 (7)	4·2 (5)
C(24)	0·5996 (7)	−0·1484 (9)	0·0342 (6)	3·7 (5)
C(25)	0·7996 (7)	−0·2234 (10)	0·0955 (6)	3·5 (4)
C(26)	0·8667 (6)	−0·2081 (9)	0·1259 (6)	3·0 (4)
C(27)	0·8900 (6)	−0·2707 (10)	0·1784 (6)	3·4 (4)
C(28)	0·8461 (8)	−0·3486 (9)	0·2004 (6)	4·4 (5)
C(29)	0·7790 (7)	−0·3640 (9)	0·1700 (7)	5·5 (6)
C(30)	0·7558 (6)	−0·3014 (11)	0·1175 (7)	4·5 (5)
C(31)	0·8172 (6)	−0·1808 (10)	−0·0668 (6)	2·8 (4)
C(32)	0·7851 (6)	−0·1822 (11)	−0·1306 (7)	3·4 (4)
C(33)	0·8244 (8)	−0·2123 (12)	−0·1855 (5)	6·3 (7)
C(34)	0·8957 (8)	−0·2409 (12)	−0·1766 (6)	6·5 (7)
C(35)	0·9278 (6)	−0·2396 (11)	−0·1128 (8)	4·4 (5)
C(36)	0·8886 (7)	−0·2095 (11)	−0·0579 (5)	3·6 (5)
C(37)	0·8646 (7)	0·2714 (12)	0·1751 (6)	2·1 (4)
C(38)	0·9009 (8)	0·1897 (9)	0·2044 (8)	6·0 (6)
C(39)	0·9431 (8)	0·2042 (12)	0·2622 (8)	6·1 (7)
C(40)	0·9491 (8)	0·3004 (15)	0·2907 (7)	6·4 (7)
C(41)	0·913 (1)	0·3821 (11)	0·2614 (9)	9 (1)
C(42)	0·8706 (9)	0·3676 (10)	0·2036 (8)	7·6 (8)
C(43)	0·8647 (7)	0·3474 (10)	0·0203 (6)	2·8 (4)
C(44)	0·9146 (8)	0·3036 (8)	−0·0214 (7)	4·7 (5)
C(45)	0·9535 (7)	0·3643 (12)	−0·0645 (6)	4·8 (5)
C(46)	0·9424 (7)	0·4688 (11)	−0·0658 (6)	5·0 (6)
C(47)	0·8925 (8)	0·5126 (8)	−0·0240 (7)	5·2 (6)
C(48)	0·8536 (6)	0·4519 (10)	0·0190 (6)	4·2 (5)
C(49)	0·7166 (6)	0·3507 (9)	0·1062 (6)	2·5 (4)
C(50)	0·6931 (7)	0·3628 (10)	0·1714 (5)	3·7 (5)
C(51)	0·6351 (7)	0·4259 (11)	0·1837 (5)	5·2 (6)
C(52)	0·6006 (6)	0·4768 (10)	0·1309 (7)	4·4 (5)
C(53)	0·6240 (7)	0·4647 (10)	0·0658 (6)	4·9 (6)
C(54)	0·6320 (7)	0·4016 (10)	0·0534 (5)	3·2 (4)

* All C-atom *B*'s are isotropic.

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

Os—Br(1)	2·507 (2)	Sb(1)—C(1)	2·16 (1)
Os—Br(2)	2·508 (2)	Sb(2)—C(25)	2·13 (1)
Os—Br(3)	2·522 (2)	Sb(2)—C(19)	2·14 (1)
Os—Sb(3)	2·640 (2)	Sb(2)—C(31)	2·15 (1)
Os—Sb(1)	2·644 (2)	Sb(3)—C(37)	2·12 (1)
Os—Sb(2)	2·654 (2)	Sb(3)—C(49)	2·12 (1)
Sb(1)—C(7)	2·13 (1)	Sb(3)—C(43)	2·13 (1)
Sb(1)—C(13)	2·13 (1)		
Br(1)—Os—Br(2)	175·27 (8)	C(13)—Sb(1)—C(1)	100·8 (4)
Br(1)—Os—Br(3)	87·43 (8)	C(7)—Sb(1)—Os	117·6 (3)
Br(1)—Os—Sb(3)	87·68 (7)	C(13)—Sb(1)—Os	122·5 (3)
Br(1)—Os—Sb(1)	83·35 (7)	C(1)—Sb(1)—Os	116·1 (3)
Br(1)—Os—Sb(2)	99·50 (6)	C(25)—Sb(2)—C(19)	99·1 (5)
Br(2)—Os—Br(3)	93·67 (8)	C(25)—Sb(2)—C(31)	101·7 (5)
Br(2)—Os—Sb(3)	87·76 (6)	C(19)—Sb(2)—C(31)	102·7 (5)
Br(2)—Os—Sb(1)	95·81 (7)	C(25)—Sb(2)—Os	114·6 (3)
Br(2)—Os—Sb(2)	85·19 (6)	C(19)—Sb(2)—Os	121·0 (3)
Br(3)—Os—Sb(3)	88·04 (7)	C(31)—Sb(2)—Os	114·9 (4)
Br(3)—Os—Sb(1)	170·14 (7)	C(37)—Sb(3)—C(49)	99·3 (5)
Br(3)—Os—Sb(2)	84·69 (6)	C(37)—Sb(3)—C(43)	99·7 (5)
Sb(3)—Os—Sb(1)	95·05 (5)	C(49)—Sb(3)—C(43)	101·6 (5)
Sb(3)—Os—Sb(2)	169·52 (5)	C(37)—Sb(3)—Os	118·5 (4)
Sb(1)—Os—Sb(2)	93·38 (5)	C(49)—Sb(3)—Os	117·3 (3)
C(7)—Sb(1)—C(13)	100·2 (5)	C(43)—Sb(3)—Os	117·3 (3)
C(7)—Sb(1)—C(1)	95·0 (5)		

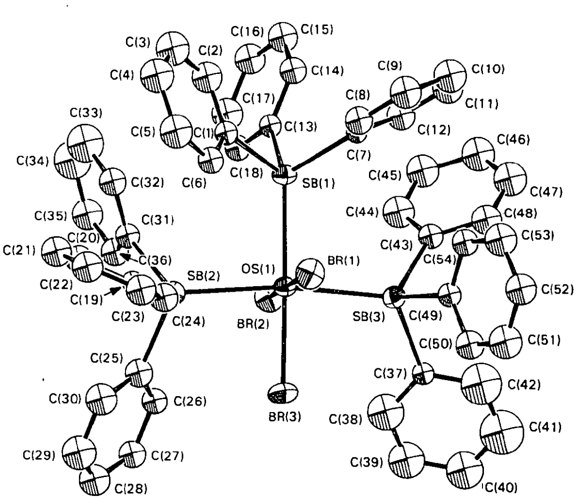

Fig. 1. Molecular structure and numbering scheme; thermal ellipsoids at the 50% probability level. Isotropic C atoms, H atoms have been omitted to improve clarity.

Direct methods provided the locations of the Os, Br and Sb atoms plus 41 of the 54 C atoms; a difference Fourier synthesis revealed the remaining non-H atoms. Full-matrix least-squares anisotropic refinement of F magnitudes using all non-H atoms revealed problems with the phenyl rings, *i.e.* one non-positive-definite C atom, a number of unusually high B_{eq} values, and generally inferior ring geometry. Problems attributed to minor ring disorder and refinement repeated using rigid-group refinement methods for the nine (C_6H_5) groups (rigid-group geometry: planar, C—C = 1·395 Å, C—C—C = 120°, C—H = 0·95 Å, C—C—H = 120°). The parameters varied for each group were the atomic coordinates of the origin atom (the C atom with no attached H atom), three angles which describe the orientation of the group relative to the crystal axial system and a group isotropic temperature factor; remaining atoms were varied anisotropically. During the final cycles of refinement the group temperature factors were omitted and the B's of the ring C atoms were varied isotropically while the H-atom B's were fixed at $1·2 \times B$ of their associated C atom. Convergence yielded $R = 0·055$, $wR = 0·059$ [$w = 1/\sigma^2(|F_o|)$], $S = 1·30$, and $(\Delta/\sigma)_{max} = 0·0007$. A final difference Fourier synthesis showed $(\Delta\rho)_{max} = 1{\cdot}41$ and $(\Delta\rho)_{min} = -1{\cdot}76$ e Å^{-3}, both located near the Os atom. Atomic scattering factors and anomalous-dispersion corrections from Cromer & Waber (1974), no extinction correction applied. Table 1 presents atomic coordinates and thermal parameters, Table 2 lists selected interatomic distances and angles and Fig. 1 illustrates the molecular conformation.* All computer programs from the *TEXSAN* crystal structure analysis package (Molecular Structure Corporation, 1985).

Related literature. This compound is one of a series prepared using the mixed solvent acetic acid/acetic anhydride (Hinckley, Matusz, Kibala & Robinson, 1987; Hinckley, Matusz & Robinson, 1988; Robinson, Hinckley, Matusz & Kibala, 1988; Robinson, Hinckley & Ikuo, 1988). It is apparently the first compound containing an Os—Sb bond to be structurally characterized.

This work was supported by the Southern Illinois University Materials Technology Center.

References

Cromer, D. T. & Waber, J. T. (1974). In *International Tables for X-ray Crystallography*, Vol. IV, p. 71 (scattering factors), p. 148 (anomalous dispersion). Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Hinckley, C. C., Matusz, M., Kibala, P. A. & Robinson, P. D. (1987). *Acta Cryst.* C**43**, 1880–1882.

Hinckley, C. C., Matusz, M. & Robinson, P. D. (1988). *Acta Cryst.* C**44**, 371–372.

Molecular Structure Corporation (1985). *TEXSAN. Structure Analysis Package.* MSC, 3304 Longmire Drive, College Station, TX 77840, USA.

Robinson, P. D., Hinckley, C. C. & Ikuo, A. (1988). *Acta Cryst.* C**44**, 1491–1492.

Robinson, P. D., Hinckley, C. C., Matusz, M. & Kibala, P. A. (1988). *Acta Cryst.* C**44**, 619–621.

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* Lists of structure factors, H-atom coordinates, anisotropic thermal parameters, intermolecular distances, and selected least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51122 (63 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 (*tert*-Butyl)$_4$As$_4$S$_4$

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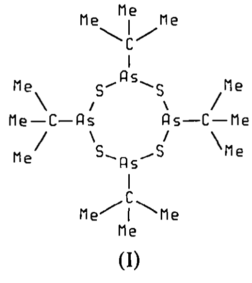
Abstract. 2,4,6,8-Tetra(*tert*-butyl)cyclotetraarsathiane, $C_{16}H_{36}As_4S_4$, $M_r = 656{\cdot}4$, monoclinic, $P2_1/c$, $a = 19{\cdot}989\,(3)$, $b = 6{\cdot}222\,(1)$, $c = 21{\cdot}938\,(4)$ Å, $\beta = 98{\cdot}88\,(1)°$, $V = 2696\,(1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}62$ g cm^{-3}, $\lambda(\text{Mo}\,K\alpha) = 0{\cdot}71073$ Å, $\mu = 52{\cdot}0$ cm^{-1}, $F(000) = 1312$, $T = 293$ K, $R = 0{\cdot}038$ for 2003 unique observed reflections. The molecule is composed of an eight-membered As_4S_4 ring with one exocyclic *tert*-butyl group bonded to each As atom. The central ring approximates a crown (S_8) conformation and the *exo* groups are equatorial. The As—S distances vary from 2·232 (3) to 2·249 (3) Å; the intra-ring angles at As average 101·5 (7) and those at S average 95·8 (15)°. The ring is flexed such that the angles between the planes of the As—S—As units and the plane of the four As atoms are 133·3 (1), 110·0 (1), 133·5 (1) and 108·9 (1)°, making the S(1)···S(3) cross-ring distance 5·374 (5) Å *vs* the S(2)···S(4) distance of 4·327 (5) Å.

Experimental. Compound prepared by the reaction of $C_4H_9PCl_2$ with Li_2S_2. Crystals of (I) obtained from benzene solutions. Colorless data crystal 0·15 × 0·20 × 0·30 mm mounted in a capillary under Ar. Intensities measured with an Enraf–Nonius CAD-4 diffractometer using ω–2θ scans of 4 to 16° min^{-1} in θ. Unit cell determined from least-squares analysis of angle data for 25 reflections with $24 < 2\theta < 32°$. Analytical absorption correction based on ψ scans varied from 0·74 to 1·00. Data collected to $(\sin\theta)/\lambda$ of 0·60 Å^{-1}, $0 < h < 23$, $-7 < k < 0$, $-26 < l < 26$. Three standard reflections $(4,0,\overline{14}$, $11,\bar{1},\bar{5}$, $10,\bar{2},1)$ decreased 5·9% over 60·1 h of data collection. 4871 reflections measured, 4726 unique ($R_{int} = 0{\cdot}02$), 2723 reflections with $I < 3\sigma(I)$ considered unobserved.



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