

Table 2. Selected geometric parameters (Å, °)

Sn—C11	2.611 (1)	N15—C15	1.431 (7)	
Sn—C12	2.618 (1)	N21—C21	1.479 (7)	
Sn—C1	2.121 (5)	N21—C22	1.334 (6)	
O151—N15	1.208 (8)	N21—C25	1.374 (6)	
O152—N15	1.218 (8)	N23—C22	1.334 (6)	
N11—C11	1.473 (7)	N23—C24	1.377 (7)	
N11—C12	1.356 (7)	C12—C22	1.459 (7)	
N11—C15	1.364 (6)	C14—C15	1.355 (8)	
N13—C12	1.323 (7)	C24—C25	1.331 (8)	
N13—C14	1.361 (7)			
C11—Sn—C11'	180.0	C21—N21—C22	125.8 (5)	
C11—Sn—C12	87.5 (4)	C21—N21—C25	125.7 (4)	
C11—Sn—C12'	92.5 (4)	C22—N21—C25	108.5 (4)	
C11—Sn—C1	88.4 (2)	C22—N23—C24	109.0 (4)	
C11—Sn—C1'	91.6 (2)	N11—C12—N13	113.2 (4)	
C12—Sn—C12'	180.0	N11—C12—C22	123.1 (4)	
C12—Sn—C1	91.0 (2)	N13—C12—C22	123.5 (4)	
C12—Sn—C1'	89.0 (2)	N13—C14—C15	109.5 (5)	
C1—Sn—C1'	180.0	N11—C15—N15	123.9 (4)	
C11—N11—C12	126.6 (4)	N11—C15—C14	108.3 (4)	
C11—N11—C15	129.1 (4)	N15—C15—C14	127.9 (5)	
C12—N11—C15	104.4 (4)	N21—C22—N23	107.7 (4)	
C12—N13—C14	104.7 (4)	N21—C22—C12	126.9 (4)	
O151—N15—O152	124.3 (5)	N23—C22—C12	125.1 (4)	
O151—N15—C15	119.5 (5)	N23—C24—C25	106.9 (5)	
O152—N15—C15	116.2 (6)	N21—C25—C24	108.0 (5)	
D—H...A	D—H	H...A	D...A	D—H...A
N23—H23...C12''	0.76 (7)	2.52 (7)	3.191 (5)	147 (6)

Symmetry codes: (i) $-x, -y, -z$; (ii) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$.

The Sn and Cl atoms were located in a Patterson map. The remaining atoms (including H atoms) were obtained from difference Fourier syntheses. Refinement was by full-matrix least-squares methods with anisotropic displacement parameters for non-H atoms and isotropic displacement parameters for H atoms, B_{iso} being fixed at 4.0 \AA^2 and not refined.

Data collection: *CAD-4 software* (Enraf-Nonius, 1989). Cell refinement: *CAD-4 software*. Data reduction: *SDP-VAX* (Frenz, 1986). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1986). Program(s) used to refine structure: *SDP-VAX*. Molecular graphics: *PLATON* (Spek, 1990), *SCHAKAL* (Keller, 1988).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1160). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bromotriphenyltin–Triphenylphosphine Oxide (1/1)

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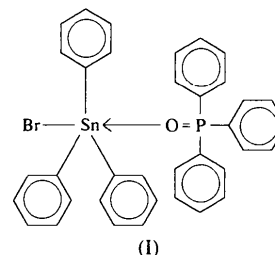
(Received 28 March 1995; accepted 5 June 1995)

Abstract

The Sn atom in bromo(triphenyl)(triphenylphosphine oxide-*O*)tin, $[\text{SnBr}(\text{C}_6\text{H}_5)_3(\text{C}_{18}\text{H}_{15}\text{OP})]$, is five-coordinate in a *trans*-trigonal bipyramidal environment.

Comment

Unlike the isomorphous pair of triphenylphosphine oxide complexes of triphenyltin chloride (Ng & Kumar Das, 1992) and triphenyllead bromide (Epply, Ealy, Yoder, Spencer & Rheingold, 1992), the isoelectronic triphenyltin bromide complex, (I), crystallizes in a polar space group, which permitted the determination of its absolute structure. The five-coordinate complex adopts



the common *trans*-trigonal bipyramidal geometry at the Sn atom.

The Sn—Br bond [2.6186 (11) Å] is lengthened relative to the bond [2.295 (2) Å] in the parent Lewis acid (Preut & Huber, 1979); the distance exceeds that [2.574 (1) Å] found in the ethylenebis(diphenylphosphine oxide) adduct of bis(4-chlorophenyl)(4-tolyl)tin bromide (Chen, Ng, Kumar Das, Jameson & Butcher, 1990). The Sn—O bond distance [2.336 (4) Å] is similar to those found in triphenylphosphine oxide complexes of other triaryltin halides, such as triphenyltin chloride [2.391 (4) Å] (Ng & Kumar Das, 1992) and tri-3-thienyltin bromide [2.335 (4) Å] (Allen, Derbyshire, Nowell & Brooks, 1984). High displacement parameters cause the bonds C3—C4 and C15—C16 to appear unrealistically short.

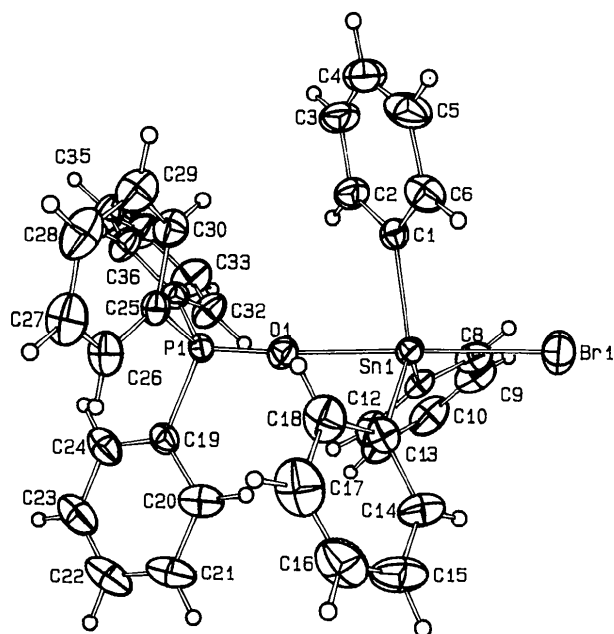


Fig. 1. Atomic labelling scheme for the title compound. Ellipsoids are drawn at the 25% probability level.

Experimental

Equimolar amounts of triphenyltin bromide and triphenylphosphine oxide were dissolved in hot ethanol; slow cooling of the filtered solution yielded large crystals of the adduct.

Crystal data

[SnBr(C₆H₅)₃(C₁₈H₁₅OP)]

$M_r = 708.17$

Monoclinic

Cc

$a = 13.200 (3) \text{ \AA}$

$b = 15.0884 (13) \text{ \AA}$

$c = 16.570 (5) \text{ \AA}$

$\beta = 105.869 (10)^\circ$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 14.5\text{--}15.0^\circ$

$\mu = 2.140 \text{ mm}^{-1}$

$T = 298 (2) \text{ K}$

Irregular

$V = 3174.4 (11) \text{ \AA}^3$

$Z = 4$

$D_x = 1.482 \text{ Mg m}^{-3}$

Data collection

Enraf–Nonius CAD-4 diffractometer

ω - 2θ scans

Absorption correction:

ψ scan (North, Phillips & Mathews, 1968)

$T_{\min} = 0.843$, $T_{\max} = 1.000$

7507 measured reflections

7247 independent reflections

$0.58 \times 0.51 \times 0.43 \text{ mm}$

Transparent

5956 observed reflections

$[I > 2\sigma(I)]$

$R_{\text{int}} = 0.0144$

$\theta_{\max} = 27.48^\circ$

$h = -17 \rightarrow 17$

$k = 0 \rightarrow 19$

$l = -21 \rightarrow 21$

3 standard reflections

frequency: 60 min

intensity decay: 3.9%

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.0440$

$wR(F^2) = 0.1034$

$S = 1.060$

7247 reflections

359 parameters

H atoms riding

$w = 1/[\sigma^2(F_o^2) + (0.0691P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.008$

$\Delta\rho_{\max} = 0.534 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.734 \text{ e \AA}^{-3}$

Extinction correction: none

Atomic scattering factors

from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Absolute configuration:

Flack (1983) parameter

= 0.035 (13)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$U_{\text{eq}} = (1/3)\sum_i U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Sn1	0.00000	-0.23931 (2)	0.00000	0.04783 (10)
Br1	0.01148 (9)	-0.17715 (6)	0.14979 (7)	0.0910 (3)
P1	0.00969 (12)	-0.32650 (9)	-0.21318 (9)	0.0475 (3)
O1	-0.0108 (3)	-0.2916 (3)	-0.1348 (3)	0.0602 (10)
C1	0.0657 (4)	-0.3650 (4)	0.0416 (4)	0.0523 (13)
C2	0.0222 (6)	-0.4423 (4)	0.0020 (6)	0.075 (2)
C3	0.0663 (9)	-0.5222 (5)	0.0263 (6)	0.095 (3)
C4	0.1524 (10)	-0.5324 (6)	0.0857 (8)	0.105 (3)
C5	0.2004 (7)	-0.4604 (8)	0.1264 (7)	0.112 (4)
C6	0.1555 (6)	-0.3744 (5)	0.1053 (5)	0.080 (2)
C7	-0.1671 (4)	-0.2269 (3)	-0.0445 (4)	0.0536 (13)
C8	-0.2283 (6)	-0.2462 (4)	0.0087 (6)	0.074 (2)
C9	-0.3385 (7)	-0.2428 (5)	-0.0220 (9)	0.102 (3)
C10	-0.3852 (7)	-0.2185 (7)	-0.1038 (8)	0.106 (4)
C11	-0.3257 (7)	-0.1978 (7)	-0.1551 (6)	0.098 (3)
C12	-0.2164 (6)	-0.2028 (6)	-0.1258 (5)	0.076 (2)
C13	0.0996 (4)	-0.1403 (4)	-0.0272 (4)	0.0547 (13)
C14	0.0739 (7)	-0.0522 (5)	-0.0235 (7)	0.096 (3)
C15	0.1375 (10)	0.0128 (6)	-0.0442 (8)	0.121 (4)
C16	0.2197 (10)	-0.0097 (9)	-0.0694 (8)	0.120 (4)
C17	0.2486 (8)	-0.0948 (11)	-0.0742 (8)	0.123 (4)
C18	0.1862 (6)	-0.1626 (6)	-0.0520 (6)	0.092 (2)
C19	-0.0206 (5)	-0.2430 (4)	-0.2933 (4)	0.0576 (14)
C20	-0.0033 (6)	-0.1530 (5)	-0.2693 (6)	0.083 (2)
C21	-0.0239 (7)	-0.0901 (6)	-0.3294 (7)	0.103 (3)
C22	-0.0618 (8)	-0.1128 (9)	-0.4137 (7)	0.113 (4)
C23	-0.0813 (7)	-0.1987 (9)	-0.4385 (6)	0.107 (3)
C24	-0.0599 (7)	-0.2657 (6)	-0.3776 (5)	0.083 (2)
C25	0.1456 (5)	-0.3575 (4)	-0.1993 (4)	0.0555 (13)
C26	0.2102 (6)	-0.3117 (6)	-0.2375 (5)	0.077 (2)
C27	0.3146 (6)	-0.3358 (7)	-0.2225 (7)	0.096 (3)
C28	0.3527 (7)	-0.4059 (7)	-0.1709 (6)	0.101 (3)
C29	0.2907 (7)	-0.4505 (6)	-0.1306 (6)	0.089 (2)
C30	0.1886 (6)	-0.4275 (6)	-0.1454 (5)	0.073 (2)

C31	-0.0708 (4)	-0.4209 (4)	-0.2522 (4)	0.0520 (12)
C32	-0.1756 (6)	-0.4133 (5)	-0.2574 (6)	0.085 (2)
C33	-0.2432 (7)	-0.4835 (6)	-0.2875 (8)	0.105 (3)
C34	-0.2055 (7)	-0.5601 (6)	-0.3153 (7)	0.093 (3)
C35	-0.1002 (7)	-0.5666 (5)	-0.3109 (6)	0.090 (2)
C36	-0.0339 (5)	-0.4990 (5)	-0.2788 (5)	0.073 (2)

Table 2. Selected geometric parameters (\AA , $^\circ$)

Sn1—C13	2.118 (6)	C14—C15	1.394 (13)
Sn1—C1	2.120 (6)	C15—C16	1.31 (2)
Sn1—C7	2.134 (6)	C16—C17	1.35 (2)
Sn1—O1	2.336 (4)	C17—C18	1.424 (15)
Sn1—Br1	2.6186 (11)	C19—C24	1.393 (12)
P1—O1	1.493 (4)	C19—C20	1.416 (10)
P1—C31	1.789 (6)	C20—C21	1.349 (11)
P1—C19	1.794 (6)	C21—C22	1.39 (2)
P1—C25	1.807 (6)	C22—C23	1.36 (2)
C1—C6	1.362 (10)	C23—C24	1.402 (13)
C1—C2	1.384 (9)	C25—C26	1.379 (9)
C2—C3	1.351 (11)	C25—C30	1.399 (10)
C3—C4	1.293 (14)	C26—C27	1.380 (11)
C4—C5	1.34 (2)	C27—C28	1.368 (14)
C5—C6	1.430 (13)	C28—C29	1.366 (14)
C7—C12	1.375 (10)	C29—C30	1.348 (11)
C7—C8	1.379 (10)	C31—C32	1.367 (9)
C8—C9	1.404 (12)	C31—C36	1.392 (9)
C9—C10	1.38 (2)	C32—C33	1.387 (11)
C10—C11	1.34 (2)	C33—C34	1.386 (13)
C11—C12	1.393 (11)	C34—C35	1.375 (12)
C13—C18	1.359 (10)	C35—C36	1.355 (10)
C13—C14	1.378 (10)		
C13—Sn1—C1	118.5 (2)	C18—C13—C14	119.3 (7)
C13—Sn1—C7	120.5 (2)	C18—C13—Sn1	120.8 (5)
C1—Sn1—C7	118.9 (2)	C14—C13—Sn1	119.8 (5)
C13—Sn1—O1	85.0 (2)	C13—C14—C15	119.8 (9)
C1—Sn1—O1	85.5 (2)	C16—C15—C14	120.2 (10)
C7—Sn1—O1	85.0 (2)	C15—C16—C17	122.7 (9)
C13—Sn1—Br1	94.1 (2)	C16—C17—C18	118.4 (10)
C1—Sn1—Br1	95.7 (2)	C13—C18—C17	119.6 (10)
C7—Sn1—Br1	94.7 (2)	C24—C19—C20	120.3 (7)
O1—Sn1—Br1	178.73 (11)	C24—C19—P1	121.1 (5)
O1—P1—C31	111.3 (3)	C20—C19—P1	118.6 (6)
O1—P1—C19	109.8 (3)	C21—C20—C19	118.9 (9)
C31—P1—C19	107.6 (3)	C20—C21—C22	120.8 (9)
O1—P1—C25	113.0 (3)	C23—C22—C21	121.6 (8)
C31—P1—C25	108.1 (3)	C22—C23—C24	119.0 (10)
C19—P1—C25	106.8 (3)	C19—C24—C23	119.4 (9)
P1—O1—Sn1	166.5 (3)	C26—C25—C30	118.5 (6)
C6—C1—C2	116.2 (6)	C26—C25—P1	122.0 (5)
C6—C1—Sn1	122.4 (5)	C30—C25—P1	119.5 (5)
C2—C1—Sn1	121.3 (5)	C27—C26—C25	119.9 (8)
C3—C2—C1	121.4 (8)	C28—C27—C26	119.7 (9)
C4—C3—C2	123.4 (9)	C29—C28—C27	121.3 (8)
C3—C4—C5	118.8 (9)	C30—C29—C28	119.2 (9)
C4—C5—C6	120.3 (9)	C29—C30—C25	121.4 (8)
C1—C6—C5	119.9 (8)	C32—C31—C36	119.2 (6)
C12—C7—C8	118.6 (6)	C32—C31—P1	116.5 (5)
C12—C7—Sn1	121.9 (5)	C36—C31—P1	124.3 (4)
C8—C7—Sn1	119.4 (5)	C31—C32—C33	119.9 (7)
C7—C8—C9	119.4 (9)	C34—C33—C32	120.2 (7)
C10—C9—C8	120.5 (10)	C35—C34—C33	119.4 (7)
C11—C10—C9	120.2 (8)	C36—C35—C34	120.2 (7)
C10—C11—C12	119.7 (9)	C35—C36—C31	121.1 (6)
C7—C12—C11	121.5 (8)		

H atoms were allowed to ride on their parent C atoms with $U(\text{H}) = 1.5U_{\text{eq}}$ of the parent atom.

Data collection: *CAD-4 VAX/PC* (Enraf-Nonius, 1988). Cell refinement: *CAD-4 VAX/PC*. Data reduction: *Xtal3.0* (Hall & Stewart, 1990). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1056). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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exo- π -(Benzonorbornadiene)tricarbonylchromium

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Abstract

The title compound, tricarbonyl[(4a,5,6,7,8,8a- η)-1,4-dihydro-1,4-methanonaphthalene]chromium, $[\text{Cr}(\text{C}_{11}\text{H}_{10})\text{(CO)}_3]$, is a complex of chromium(0) in which the arene ring of the organic ligand is hexahapto-coordinated to the Cr atom. Three terminal carbonyl ligands form the triangular base of the molecule. The mode of coordination of the $\text{Cr}(\text{CO})_3$ moiety to the ligand is such