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Structure of Triphenyltin Bromide

BY HANS PREUT AND FRIEDO HUBER*

Lehrstuhl für Anorganische Chemie II der Universität, D-4600 Dortmund 50, Federal Republic of Germany

Abstract. $(C_6H_5)_3SnBr$, $M_r = 429.92$, monoclinic, $P2_1/c$, $a = 19.082$ (3), $b = 9.763$ (2), $c = 18.739$ (3) Å, $\beta = 106.32$ (8)°, $Z = 8$, $V = 3350.3$ Å^3, μ(Mo $K\alpha$) = 3.73 mm^{-1}, $D_c = 1.704$ Mg m^{-3}, $R = 0.052$. The asymmetric unit contains two slightly distorted tetrahedral molecules. The mean parameters are: Sn—Br = 2.495 (2), Sn—C = 2.114 (8) Å; ∠Br—Sn—C = 105.2 (2), ∠C—Sn—C = 113.4 (3)°.

Introduction. We have recently reported the structures of Ph$_3$PbX (Ph = C$_6$H$_5$, X = Cl, Br) (Preut & Huber, 1977) and Ph$_3$GeBr (Preut & Huber, 1979). As part of our study of coordinative and structural correlations in organocompounds of Group IVb elements we have determined the crystal structure of Ph$_3$SnBr. Data were collected with a crystal 0.34 × 0.23 × 0.18 mm.

Cell parameters were determined by least squares from 23 reflections on a Hilger & Watts Y290 automatic four-circle diffractometer with graphite-monochromatized Mo $K\alpha$ radiation and a scintillation counter. The intensities of 3988 reflections ($I > 3\sigma$) with $2 \leq \theta \leq 28.1°$ were measured by the $\omega/2\theta$ scan technique, with a scan width $\Delta 2\theta = (1.34 + 0.34 \tan \theta)°$ from background to background and a scan speed of 0.02° s^{-1} in 2θ. Backgrounds were measured at each end of the scan range for 7 s. Five standard reflections were measured every fifty reflections during data collection and showed only random deviations. Lorentz and polarization corrections were applied, but no corrections were made for absorption. After averaging the equivalent reflections the data set contained 3292 unique reflections. The structure was solved by Patterson and Fourier methods and refined

by blocked-full-matrix least squares with *SHELX* (Sheldrick, 1976). During the refinement the phenyl rings were treated as rigid hexagons (∠C—C—C = 120°; C—C = 1.395, C—H = 1.080 Å) with individual isotropic temperature factors for the C atoms and one common temperature factor for the H atoms of one molecule. In the final stages of refinement, each reflection was assigned a weight $w^{-1} = \sigma^2(F_o) + 0.005F_o^2$ and reflections with $F_o < 4\sigma(F_o)$ were omitted (757 reflections suppressed out of 3292). The refinement converged to $R = \sum \Delta / \sum F_o = 0.052$ and $R_w = (\sum w^{1/2} \Delta / \sum w^{1/2} F_o) = 0.052$ for 75 refined parameters.

The final coordinates are listed in Table 1 and bond distances and angles in Table 2. Fig. 1 shows the two molecules in the asymmetric unit, and the numbering of the atoms.†

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† Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34090 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
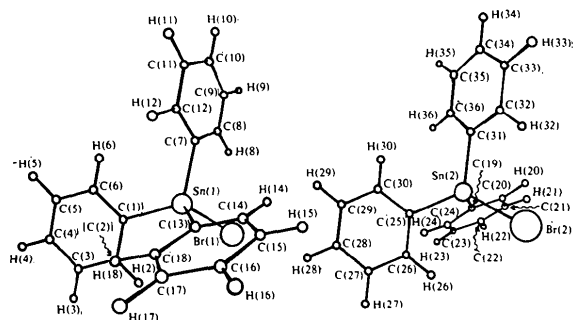
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* To whom correspondence should be addressed.

Fig. 1. The two molecules of $(C_6H_5)_3SnBr$ in the asymmetric unit, showing the atom-numbering scheme.

Table 1. *Final atomic positional parameters* (×10⁴) *with e.s.d.'s in parentheses*

	x	y	z		x	y	z
Sn(1)	464 (1)	572 (1)	1714 (1)	H(17)	342 (4)	−4406 (8)	680 (5)
Sn(2)	5527 (1)	1880 (1)	1905 (1)	H(18)	−171 (4)	−2217 (8)	971 (5)
Br(1)	931 (1)	2440 (2)	1066 (1)	C(19)	5940 (4)	3673 (8)	1541 (4)
Br(2)	6024 (1)	−97 (2)	1352 (1)	C(20)	6691 (4)	3908 (8)	1754 (4)
C(1)	−678 (4)	856 (10)	1412 (4)	C(21)	6969 (4)	5119 (8)	1546 (4)
C(2)	−1072 (4)	1066 (10)	671 (4)	C(22)	6495 (4)	6095 (8)	1125 (4)
C(3)	−1827 (4)	1246 (10)	482 (4)	C(23)	5744 (4)	5860 (8)	912 (4)
C(4)	−2188 (4)	1215 (10)	1034 (4)	C(24)	5466 (4)	4649 (8)	1120 (4)
C(5)	−1794 (4)	1005 (10)	1775 (4)	H(20)	7058 (4)	3153 (8)	2080 (4)
C(6)	−1039 (4)	825 (10)	1963 (4)	H(21)	7550 (4)	5301 (8)	1710 (4)
H(2)	−792 (4)	1090 (10)	244 (4)	H(22)	6710 (4)	7032 (8)	964 (4)
H(3)	−2132 (4)	1409 (10)	−90 (4)	H(23)	5377 (4)	6615 (8)	586 (4)
H(4)	−2773 (4)	1355 (10)	888 (4)	H(24)	4884 (4)	4467 (8)	955 (4)
H(5)	−2074 (4)	981 (10)	2202 (4)	C(25)	4383 (4)	1649 (10)	1483 (4)
H(6)	−734 (4)	662 (10)	2537 (4)	C(26)	4076 (4)	1413 (10)	725 (4)
C(7)	956 (5)	903 (8)	2864 (4)	C(27)	3322 (4)	1268 (10)	440 (4)
C(8)	1269 (5)	2159 (8)	3135 (4)	C(28)	2874 (4)	1359 (10)	912 (4)
C(9)	1579 (5)	2342 (8)	3897 (4)	C(29)	3181 (4)	1594 (10)	1670 (4)
C(10)	1576 (5)	1270 (8)	4388 (4)	C(30)	3935 (4)	1739 (10)	1956 (4)
C(11)	1263 (5)	15 (8)	4117 (4)	H(26)	4423 (4)	1343 (10)	359 (4)
C(12)	953 (5)	−168 (8)	3355 (4)	H(27)	3084 (4)	1086 (10)	−146 (4)
H(8)	1271 (5)	2989 (8)	2754 (4)	H(28)	2290 (4)	1246 (10)	691 (4)
H(9)	1822 (5)	3314 (8)	4106 (4)	H(29)	2835 (4)	1664 (10)	2036 (4)
H(10)	1816 (5)	1412 (8)	4978 (4)	H(30)	4173 (4)	1922 (10)	2543 (4)
H(11)	1261 (5)	−815 (8)	4497 (4)	C(31)	5948 (5)	1628 (8)	3073 (5)
H(12)	711 (5)	−1140 (8)	3145 (4)	C(32)	6286 (5)	414 (8)	3381 (5)
C(13)	876 (4)	−1254 (8)	1392 (5)	C(33)	6551 (5)	276 (8)	4151 (5)
C(14)	1630 (4)	−1406 (8)	1537 (5)	C(34)	6477 (5)	1354 (8)	4613 (5)
C(15)	1920 (4)	−2639 (8)	1373 (5)	C(35)	6139 (5)	2568 (8)	4306 (5)
C(16)	1455 (4)	−3721 (8)	1064 (5)	C(36)	5874 (5)	2705 (8)	3536 (5)
C(17)	701 (4)	−3569 (8)	920 (5)	H(32)	6343 (5)	−420 (8)	3022 (5)
C(18)	412 (4)	−2335 (8)	1084 (5)	H(33)	6813 (5)	−663 (8)	4389 (5)
H(14)	1989 (4)	−569 (8)	1776 (5)	H(34)	6683 (5)	1248 (8)	5209 (5)
H(15)	2503 (4)	−2757 (8)	1485 (5)	H(35)	6082 (5)	3403 (8)	4664 (5)
H(16)	1680 (4)	−4676 (8)	937 (5)	H(36)	5612 (5)	3646 (8)	3297 (5)

Table 2. *Bond distances* (Å) *and angles* (°) *in* Ph₃SnBr

Sn(1)−Br(1)	2·490 (2)	Sn(1)−C(13)	2·104 (8)
Sn(2)−Br(2)	2·500 (2)	Sn(2)−C(19)	2·110 (8)
Sn(1)−C(1)	2·112 (8)	Sn(2)−C(25)	2·114 (8)
Sn(1)−C(7)	2·119 (7)	Sn(2)−C(31)	2·123 (8)
Sn−Br(mean)	2·495 (2)	Sn−C(mean)	2·114 (8)
Br(1)−Sn(1)−C(1)	104·9 (2)	C(1)−Sn(1)−C(7)	112·5 (3)
Br(1)−Sn(1)−C(7)	105·5 (2)	C(1)−Sn(1)−C(13)	118·3 (3)
Br(1)−Sn(1)−C(13)	105·5 (2)	C(7)−Sn(1)−C(13)	109·0 (3)
Br(2)−Sn(2)−C(19)	106·7 (2)	C(19)−Sn(2)−C(25)	114·1 (3)
Br(2)−Sn(2)−C(25)	103·6 (2)	C(19)−Sn(2)−C(31)	111·2 (3)
Br(2)−Sn(2)−C(31)	105·1 (2)	C(25)−Sn(2)−C(31)	115·1 (3)
Br−Sn−C(mean)	105·2 (2)	C−Sn−C(mean)	113·4 (3)

Discussion. The structure of Ph₃SnBr is isotypic with Ph₃GeBr (Preut & Huber, 1979). The two molecules of the asymmetric unit differ very little in the bond angles at the Sn atom and the orientation of the phenyl groups. Sn is four-coordinated. As in Ph₃GeBr (Preut & Huber, 1979) and Ph₃SnCl, which is structurally analogous (Bokii, Zacharova & Struchkov, 1970), the mean C−Sn−C angles are greater, and the C−Sn−Br angles smaller than the ideal tetrahedral angle. The Sn−Br distances correspond to those in other Snᴵⱽ compounds: in (4-bromo-1,2,3,4-tetraphenyl-1,3-butadienyl)dimethyltin bromide 2·504 (5) Å, in SnBr₄ 2·44 (2) Å (Lister & Sutton, 1941), and in (CH₃)₄₋ₙSnBrₙ 2·49 (3) (n = 1), 2·48 (n = 2) and 2·45 Å (n = 3, by electron diffraction) (Skinner & Sutton, 1944).

The shortest distances between Br and the Sn atom of a neighbouring molecule are 5·925 (2) and 5·792 (2) Å [Sn(1)···Br(1) and Sn(2)···Br(2)], and therefore coordination can be excluded. In this respect Ph₃SnBr differs from the analogous compound of Pb and shows the same behaviour as the lighter Ge. In Ph₃PbBr the five-coordinated Pb atoms are bridged by Br and the Ph₃Pb moieties are nearly planar (Preut & Huber,

Table 3. *Planes, distances* (Å) *of* Sn *therefrom and dihedral angles* (°)

Planes are defined in terms of Cartesian coordinates by $Ax + By + Cz = D$; x is parallel to a, y is in the plane ab, and z is parallel to c^*; A, B, and C are the direction cosines of the normal to the plane and D is the distance of the plane from the origin. The values for A, B, and C are multiplied by 10^4.

Plane		A	B	C	D (Å $\times 10^2$)	Distance of Sn from plane (Å $\times 10^2$)
(I)	C(1) to H(6)	−974	−9872	−1259	−78·5	0·8 (1)
(II)	C(7) to H(12)	9449	−3147	−901	−44·3	−2·7 (1)
(III)	C(13) to H(18)	2121	3372	−9172	−251·1	−13·2 (1)
(IV)	C(19) to H(24)	3193	−4387	−8400	−54·2	−9·4 (1)
(V)	C(25) to H(30)	−445	9861	−1602	−82·3	1·4 (1)
(VI)	C(31) to H(36)	9305	3649	−317	−946·0	−1·9 (1)
(VII)	Sn(1), Br(1), C(1)	1317	−5958	−7923	−277·9	0·0
(VIII)	Sn(1), Br(1), C(7)	8155	−5773	−412	−46·3	0·0
(IX)	Sn(1), Br(1), C(13)	−6209	−794	−7799	−243·9	0·0
(X)	Sn(2), Br(2), C(19)	−5944	321	−8036	−836·7	0·0
(XI)	Sn(2), Br(2), C(25)	2412	5619	−7913	−62·3	0·0
(XII)	Sn(2), Br(2), C(31)	8389	5441	−114	−896·7	0·0

The sequence of atoms in planes I to VI is according to Table 1.

Dihedral angles

∠(I)/(VII)	47·5	∠(II)/(VIII)	17·1
∠(III)/(IX)	56·2	∠(V)/(XI)	47·9
∠(VI)/(XII)	11·6	∠(IV)/(X)	61·9

1977). Details of the least-squares planes in Ph$_3$SnBr are given in Table 3.

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Dibenzylmercury

By Peter B. Hitchcock

School of Molecular Sciences, University of Sussex, Brighton BN1 9QJ, England

Abstract. $C_{14}H_{14}Hg$, $M_r = 382·9$, tetragonal, $P4_2/n$, $a = 12·881$ (5), $c = 7·076$ (3) Å, $U = 1174·1$ Å^3, $Z = 4$, $D_c = 2·17$ Mg m^{-3}, $F(000) = 712$. Mo $K\alpha$ radiation, $\mu = 12·59$ mm^{-1}. $R_F = 0·044$ for 586 counter reflections. The molecule lies on a crystallographic twofold axis and has linear coordination at Hg, with Hg–C 2·07 (2) Å, and no abnormal intramolecular contacts.

Introduction. Studies of the thermal decomposition of dibenzylmercury (Jackson & O'Neill, 1978) raise the possibility that there may be some interaction of the Hg atom with the phenyl ring as in Zr and Hf benzyls (Davies, Jarvis & Kilbourn, 1971). The structure of benzyl(triphenylmethylthio)mercury (Bach, Weibel, Schmonsees & Glick, 1974) shows no such interaction