

Synthesis and crystal structure of bis(triphenylarsine oxide)(*p*-chlorophenyl)diphenyltin(IV) tetraphenylborate, $[(p\text{-ClC}_6\text{H}_4)\text{Ph}_2\text{Sn}(\text{Ph}_3\text{AsO})_2][\text{BPh}_4]$

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Abstract

The crystal structure of the mixed-aryl triorganotin cationic complex, $[(p\text{-ClC}_6\text{H}_4)\text{Ph}_2\text{Sn}(\text{Ph}_3\text{AsO})_2]^+$, isolated as the tetraphenylborate salt, has been determined. The compound crystallises in the space group $P2_1/c$ with a 9.698(3), b 21.259(5), c 32.672(7) Å; β 101.32(2)°; V 6605(5) Å³; $Z = 4$; the unit cell comprises four non-interacting ion pairs. The structure was refined to $R = 0.07$ for 5316 observed Mo-K_α reflections. The coordination environment of tin is a near-ideal trigonal bipyramid with the *ipso*-carbons of the aryl rings lying in the equatorial plane and the axial positions being occupied by the oxygen atoms of the Ph_3AsO ligands. The geometry of the tetraphenylborate anion is unequivocally tetrahedral.

Introduction

Trigonal bipyramidal coordination is a well-documented structural feature among five-coordinate triorganotin(IV) compounds [1,2], the only exception known being for the recently reported tribenzyl(2-thiolatopyridine-*N*-oxide)tin, which has a square-pyramidal geometry [3]. A favoured stereochemical configuration in the trigonal bipyramidal structures is the equatorial array of the three tin-bound organic groups. We have previously assigned this configuration on the basis of spectral evidence to the cationic complexes of the formulation $[\text{R}_3\text{SnL}_2]^+[\text{BPh}_4]^-$, involving neutral, monodentate, oxygen-donor ligands [4–6]. For the corresponding complexes involving neutral chelating ligands (Ch), $[\text{R}_3\text{Sn}(\text{Ch})]^+[\text{BPh}_4]^-$, a meridional R_3Sn skeletal configuration was inferred from their tin-119m Mössbauer spectra [6].

Although solution studies on the above complexes were generally thwarted by solubility problems, ^1H NMR data secured in CDCl_3 for the few cases among the bis(monodentate) complexes revealed that strong donor ligands such as Ph_3AsO permitted retention of stereochemical integrity in solution, but with weaker ligands such as DMSO or DMF, a facile phenyl group transfer from $[\text{BPh}_4]^-$ to tin occurred, leading to the formation R_3SnPh [4]. This suggested that the complexes existed as ion-pairs in solution, and possibly also in the solid state.

A more intimate interaction between the metal atom and the counterion, however, has been reported [7,8] in complexes of $[\text{BPh}_4]^-$ with rhodium(I) and iridium(I), namely the engagement of one of the phenyl groups of the tetraphenylborate anion in $\pi(\eta^6)$ -bonded interaction with the transition metal, as crystallographically evidenced for $\{\text{Rh}[\text{P}(\text{OMe})_3]_2\}^+[\text{BPh}_4]^-$ (metal to mean plane of ring distance, 1.86 Å) [8]. Although a similar π -bonded interaction is unlikely in tetraphenylborate complexes of tin, we were nevertheless interested in studying the crystal structure of the mixed aryl triorganotin cationic complex, $[(p\text{-ClC}_6\text{H}_4)\text{Ph}_2\text{Sn}(\text{Ph}_3\text{AsO})_2]^+[\text{BPh}_4]^-$, to examine the effects of the steric bulk of the ligands on the overall coordination geometry at tin, and also the role, if any, played by the counterion in stabilising the structure.

Experimental

Preparation of title complex (I). Solutions of $(p\text{-ClC}_6\text{H}_4)\text{Ph}_2\text{SnBr}$ (0.2 g, 0.4 mmol), Ph_3AsO (0.25 g, 0.8 mmol) and $\text{Na}[\text{BPh}_4]$ (0.14 g, 0.4 mmol) in methanol

Table 1

Data collection and processing parameters

Molecular formula	$\text{C}_{78}\text{H}_{64}\text{As}_2\text{BClO}_2\text{Sn}$
Molecular weight	1348.18
Space group	$P2_1/c$
Cell constants	a 9.698(3) Å b 21.259(5) Å c 32.672(7) Å β 101.32(2)° V 6605(5) Å ³
Z	4
D_m	1.36 (KI/ H_2O) g cm^{-3}
D_c	1.356 g cm^{-3}
Crystal size	0.04 × 0.03 × 0.02 mm (block shape)
μ	14.65 cm^{-1}
Absorption correction	max. 1.11, min. 0.78
Collection range	h : -11 to 11, k : 0 to 25, l : 0 to 38
Number of unique reflections	11703
Number of observed reflections	5316 criterion: $ F_o > 3\sigma(F_o)$
Number of variables	761
R	0.071
R_w	0.067
w	$4(F_o)^2 / [\sigma(F_o)^2]^2$
Max. shift/error	0.01
Final Fourier difference map	0.34 $\text{e}/\text{Å}^3$ maximum peak height
$F(000)$	2744

were mixed and ca. 200 ml of water were added slowly with stirring. The white solid obtained was recrystallised from methanol in 82% yield (0.45 g), m.p. 173–175°C (decomp.) Anal. Found: C, 69.23; H, 4.84. $C_{78}H_{64}As_2BClO_2Sn$ calcd.: C, 68.80; H, 4.70%. 119m Sn Mössbauer data (mm s⁻¹, 80 K): isomer shift, 1.16, quadrupole splitting 3.27; Γ_1 0.9; Γ_2 0.85.

X-ray analysis. Diffraction measurements on the title complex were made on a Nicolet P3m automatic diffractometer (graphite monochromatized Mo- K_α radiation, λ 0.71073 Å). Standard centering and auto-indexing procedures indicated a monoclinic cell which was confirmed by axial photographs. The orientation matrix and accurate unit-cell dimensions were determined from a least-squares fit of 15 reflections ($28^\circ < 2\theta < 35^\circ$) scattered evenly throughout the reciprocal space. Intensities were recorded at 23°C. Procedures for data collection were as previously described [9]. The intensities of 4 standard reflections (300, 040, 004, 115) measured after every 96 data showed no evidence of significant crystal decay during the entire data collection. Redundant and equivalent reflections were averaged and converted to unscaled $|F_0|$ values following corrections for Lorentz and polarization factors. The structure was solved by use of direct methods (MULTAN-80) which located the positions of the Sn and two As atoms. Subsequent least squares refinement and difference Fourier syntheses revealed the remaining atoms.

All non-hydrogen atoms except chlorine were refined anisotropically. The hydrogen atoms were assigned fixed isotropic temperature factors equal to 0.05 Å². All computations were performed on a PDP11/73 minicomputer with the TEXRAY program package [10]. The atomic scattering factors were taken from International Tables for X-ray Crystallography, volume IV [11]. The effects of anomalous dispersion for all non-hydrogen atoms were included in F_c [12]. The final R indices and data processing parameters are listed in Table 1. Positional and thermal parameters of the non-hydrogen atoms are given in Table 2 in accordance with the

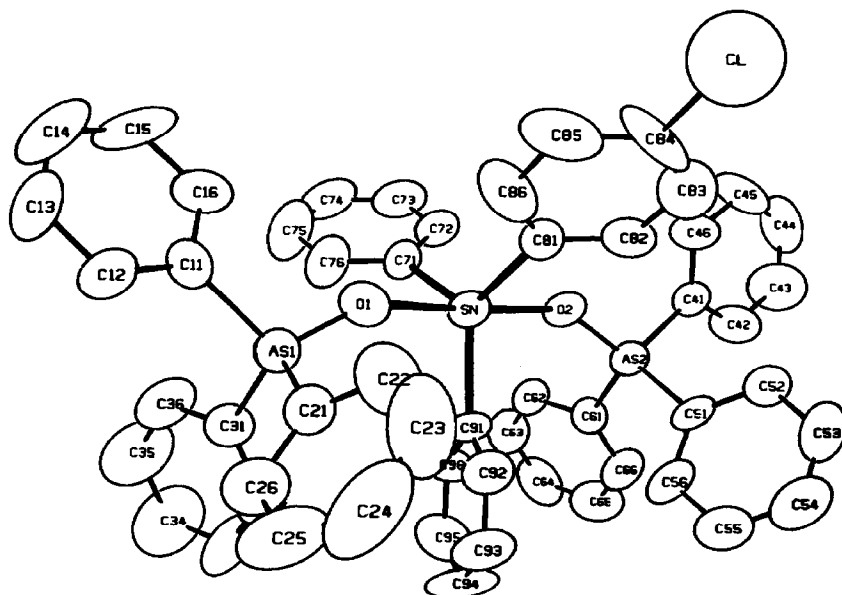


Fig. 1. Perspective view of $[(p\text{-ClC}_6\text{H}_4)\text{Ph}_2\text{Sn}(\text{Ph}_3\text{AsO})_2]^+$.

Table 2

Final atomic coordinates ($\times 10^5$ for Sn; $\times 10^4$ for other atoms) and temperature factors a ($\text{\AA}^2 \times 10^2$)

Atom	x	y	z	B_{iso} (\AA^2)
Sn	0.06521(8)	0.18923(4)	0.38298(2)	3.68(2)
As(1)	-0.0819(1)	0.33729(5)	0.41864(4)	3.97(3)
As(2)	0.0547(1)	0.02094(5)	0.35460(4)	3.67(3)
Cl	0.5752(6)	0.1451(3)	0.5486(2)	14.7(2) ^b
O(1)	0.0298(7)	0.2821(3)	0.4091(2)	4.8(2)
O(2)	0.1124(7)	0.0952(3)	0.3596(2)	4.4(2)
C(11)	0.017(1)	0.4151(5)	0.4197(3)	4.0(3)
C(12)	-0.026(1)	0.4674(5)	0.4384(4)	5.5(3)
C(13)	0.044(1)	0.5230(5)	0.4370(5)	7.3(4)
C(14)	0.157(2)	0.5275(6)	0.4192(5)	8.8(5)
C(15)	0.199(1)	0.4759(7)	0.4008(5)	8.1(4)
C(16)	0.133(1)	0.4183(6)	0.4010(4)	5.8(3)
C(21)	-0.131(1)	0.3213(5)	0.4711(4)	4.6(3)
C(22)	-0.040(1)	0.2878(6)	0.5002(4)	6.6(4)
C(23)	-0.074(2)	0.2764(6)	0.5384(4)	9.0(5)
C(24)	-0.195(2)	0.2980(7)	0.5481(4)	9.9(5)
C(25)	-0.283(1)	0.3313(7)	0.5198(4)	8.4(4)
C(26)	-0.255(1)	0.3439(6)	0.4809(4)	6.1(4)
C(31)	-0.250(1)	0.3440(5)	0.3776(4)	4.7(3)
C(32)	-0.361(1)	0.3036(6)	0.3778(4)	6.4(4)
C(33)	-0.481(1)	0.3086(6)	0.3484(5)	9.8(5)
C(34)	-0.491(2)	0.3520(8)	0.3183(6)	14.2(6)
C(35)	-0.381(2)	0.3919(8)	0.3182(5)	11.7(5)
C(36)	-0.260(1)	0.3880(6)	0.3472(4)	7.4(4)
C(41)	0.208(1)	-0.0292(5)	0.3457(3)	3.8(3)
C(42)	0.184(1)	-0.0901(6)	0.3323(4)	6.8(4)
C(43)	0.290(2)	-0.1280(7)	0.3277(6)	10.1(5)
C(44)	0.423(1)	-0.1084(6)	0.3358(5)	8.7(4)
C(45)	0.452(1)	-0.0474(7)	0.3500(5)	7.7(4)
C(46)	0.342(1)	-0.0082(5)	0.3546(4)	5.6(3)
C(51)	0.001(1)	-0.0119(5)	0.4040(3)	3.7(3)
C(52)	0.097(1)	-0.0482(6)	0.4312(4)	5.2(3)
C(53)	0.053(1)	-0.0749(6)	0.4651(4)	6.4(4)
C(54)	-0.077(1)	-0.0642(6)	0.4722(4)	7.2(4)
C(55)	-0.170(1)	-0.0289(6)	0.4459(4)	6.1(3)
C(56)	-0.132(1)	-0.0016(5)	0.4115(4)	5.2(3)
C(61)	-0.098(1)	0.0110(5)	0.3079(3)	4.1(3)
C(62)	-0.107(1)	0.0518(5)	0.2755(4)	4.7(3)
C(63)	-0.211(1)	0.0428(6)	0.2407(4)	6.5(4)
C(64)	-0.305(1)	-0.0052(6)	0.2383(4)	7.0(4)
C(65)	-0.294(1)	-0.0461(6)	0.2716(4)	6.9(4)
C(66)	-0.193(1)	-0.0380(5)	0.3057(4)	5.2(3)
C(71)	0.109(1)	0.2306(5)	0.3278(3)	3.9(3)
C(72)	0.188(1)	0.1987(5)	0.3044(3)	4.7(3)
C(73)	0.221(1)	0.2245(6)	0.2675(4)	6.6(4)
C(74)	0.174(1)	0.2847(6)	0.2564(4)	6.9(4)
C(75)	0.093(2)	0.3157(6)	0.2796(4)	7.5(4)
C(76)	0.061(1)	0.2874(5)	0.3144(4)	5.8(3)
C(81)	0.230(1)	0.1763(5)	0.4363(3)	3.8(3)
C(82)	0.280(1)	0.1163(6)	0.4484(4)	5.1(3)
C(83)	0.384(1)	0.1048(6)	0.4818(5)	7.4(4)
C(84)	0.438(1)	0.1543(7)	0.5033(4)	7.8(4)
C(85)	0.396(1)	0.2161(7)	0.4937(5)	8.0(4)
C(86)	0.289(1)	0.2248(6)	0.4600(4)	6.9(4)

Table 2 (continued)

Atom	x	y	z	B_{iso} (\AA^2)
C(91)	-0.143(1)	0.1631(5)	0.3851(4)	4.3(3)
C(92)	-0.191(1)	0.1587(5)	0.4223(4)	5.6(3)
C(93)	-0.324(1)	0.1458(6)	0.4231(4)	7.5(4)
C(94)	-0.422(1)	0.1349(6)	0.3886(6)	10.1(4)
C(95)	-0.381(1)	0.1391(6)	0.3510(4)	7.0(4)
C(96)	-0.243(1)	0.1524(5)	0.3499(4)	5.5(3)
C(111)	0.373(1)	0.3933(5)	0.6028(3)	4.3(3)
C(112)	0.427(1)	0.4554(6)	0.6034(4)	6.2(4)
C(113)	0.422(1)	0.4898(6)	0.5669(4)	7.0(4)
C(114)	0.362(1)	0.4661(7)	0.5285(4)	8.4(4)
C(115)	0.307(2)	0.4075(8)	0.5277(4)	8.6(5)
C(116)	0.311(1)	0.3720(7)	0.5631(4)	6.9(4)
C(211)	0.294(1)	0.2904(5)	0.6378(3)	4.4(3)
C(212)	0.338(1)	0.2410(6)	0.6156(4)	6.4(4)
C(213)	0.266(1)	0.1869(6)	0.6058(4)	6.7(4)
C(214)	0.142(1)	0.1767(5)	0.6193(4)	6.2(4)
C(215)	0.094(1)	0.2216(6)	0.6419(4)	6.2(3)
C(216)	0.168(1)	0.2769(5)	0.6510(3)	4.6(3)
C(311)	0.319(1)	0.3996(5)	0.6785(3)	4.3(3)
C(312)	0.187(1)	0.4276(6)	0.6662(4)	5.4(3)
C(313)	0.123(1)	0.4660(6)	0.6912(4)	6.3(4)
C(314)	0.191(1)	0.4810(6)	0.7302(4)	6.8(4)
C(315)	0.322(1)	0.4572(6)	0.7433(4)	6.9(4)
C(316)	0.383(1)	0.4182(6)	0.7193(4)	5.6(3)
C(411)	0.545(1)	0.3337(6)	0.6669(4)	5.2(3)
C(412)	0.573(1)	0.2984(6)	0.7038(4)	6.3(4)
C(413)	0.711(1)	0.2831(6)	0.7232(4)	6.5(4)
C(414)	0.825(1)	0.2993(7)	0.7043(5)	8.0(4)
C(415)	0.796(1)	0.3304(6)	0.6675(4)	7.2(4)
C(416)	0.661(1)	0.3486(6)	0.6493(4)	5.3(3)
B	0.384(1)	0.3535(6)	0.6465(4)	3.7(3)

^a Equivalent isotropic temperature factor B defined as $B_{\text{iso}} = \frac{1}{3} [a^2 B_{1,1} + b^2 B_{2,2} + c^2 B_{3,3} + ab \cos \gamma B_{1,2} + ac \cos \beta B_{1,3} + bc \cos \alpha B_{2,3}]$. ^b Atom refined isotropically.

labelling scheme of Fig. 1. Selected bond distances and bond angles are given in Table 3. Tables of hydrogen atom coordinates, least-squares planes, anisotropic temperature parameters and structure factors along with a complete list of bond distances and angles are available as supplementary material from the authors.

Results and discussion

The crystal structure consists of the packing of discrete $[(p\text{-ClC}_6\text{H}_4)\text{-Ph}_2\text{Sn}(\text{Ph}_3\text{AsO})_2]^+$ cations and tetraphenylborate anions. A perspective view of the inner coordination geometry at tin is given in Fig. 1 and a stereoscopic view of the entire complex is presented in Fig. 2.

The coordination geometry at the tin atom is a near-ideal trigonal bipyramid, with equatorial location of the *p*-chlorophenyl and two phenyl groups and axial disposition of the oxygenated ligands. The sum of the angles subtended at tin by the *ipso*-carbons of the aryl rings is 360(1)°; the corresponding axial (O–Sn–O) angle is

Table 3

Selected bond distances (Å) and angles (°) with estimated standard deviations in parentheses

<i>(a) In the tin environment</i>			
Sn–O(1)	2.205(5)	Sn–O(2)	2.219(5)
Sn–C(71)	2.122(9)	Sn–C(81)	2.138(9)
Sn–C(91)	2.108(8)		
O(1)–Sn–O(2)	176.6(2)	C(71)–Sn–C(81)	120.2(3)
C(71)–Sn–O(2)	119.3(4)	C(81)–Sn–C(91)	120.5(4)
O(1)–Sn–C(71)	91.8(3)	O(2)–Sn–C(71)	89.5(3)
O(1)–Sn–C(81)	87.0(3)	O(2)–Sn–C(81)	89.6(3)
O(1)–Sn–C(91)	90.0(3)	O(2)–Sn–C(91)	92.1(3)
Sn–C(71)–C(72)	119.9(7)	Sn–C(71)–C(76)	122.5(8)
Sn–C(81)–C(82)	120.8(7)	Sn–C(81)–C(86)	122.3(7)
Sn–C(91)–C(92)	122.3(8)	Sn–C(91)–C(96)	122.8(8)
<i>(b) In the triphenylarsine oxide ligands</i>			
As(1)–O(1)	1.667(5)	As(2)–O(2)	1.673(5)
As(1)–C(11)	1.911(9)	As(2)–C(41)	1.900(9)
As(1)–C(21)	1.899(10)	As(2)–C(51)	1.921(8)
As(1)–C(31)	1.898(9)	As(2)–C(61)	1.919(9)
O(1)–As(1)–C(11)	105.6(3)	O(2)–As(2)–C(41)	106.7(3)
O(1)–As(1)–C(21)	109.3(4)	O(2)–As(2)–C(51)	113.7(3)
O(1)–As(1)–C(31)	114.8(3)	O(1)–As(2)–C(61)	111.9(3)
C(11)–As(1)–C(21)	110.7(4)	C(41)–As(2)–C(51)	105.9(4)
C(11)–As(1)–C(31)	107.9(4)	C(41)–As(2)–C(61)	108.8(4)
C(21)–As(1)–C(31)	108.6(4)	C(51)–As(2)–C(61)	109.6(4)
Sn–O(1)–As(1)	149.2(3)	Sn–O(2)–As(2)	142.7(3)
<i>(c) In the tetraphenylborate counterion</i>			
B–C(111)	1.646(13)	B–C(211)	1.597(13)
B–C(311)	1.648(13)	B–C(411)	1.628(13)
C(111)–B–C(211)	109.7(8)	C(111)–B–C(311)	106.0(8)
C(111)–B–C(411)	112.4(8)	C(211)–B–C(311)	110.3(7)
C(211)–B–C(411)	107.7(8)	C(311)–B–C(411)	110.7(8)

176.6(2)°. The planes of the aryl rings bonded to tin are inclined by 93.5(10)° (ring C(71)–C(76)), 107.8(11)° (ring C(81)–C(86)) and 101.8(11)° (ring C(91)–C(96)) to that of the trigonal plane. The Sn–C distances (av. 2.12(1) Å) are in the range normal for pentacoordinated triaryltins [13,14] and, as expected, are longer than the As–C (av. 1.91(1) Å) and B–C (av. 1.63(1) Å) bonds (Table 3).

As is usual for complexes of Ph₃AsO, the ligand is involved in bent coordination at the metal centre. Thus the (Sn–O–As) bond angle in the present complex is 145.9(3)°, relative to 136.0° in Ph₃SnNO₃ · Ph₃AsO [15], 134.5° in (Ph₃SnCl)₂ · {Ph₂As(O)CH₂}₂ [16], 140.2° in (Ph₃SnNO₃)₂ · {Ph₂As(O)CH₂}₂ [17] and 137.9° in Ph₂Sn(NO₃)₂ · Ph₃AsO [18]. This contrasts with the case of phosphine oxide ligands (e.g. HMPA [19]), for which both linear and bent coordination have been observed. The As–O bond in the title complex, **1**, is slightly lengthened (1.670(5) Å) relative to the free ligand (1.644 Å [20]), as is the case also in the corresponding neutral 1/1 adducts cited above. This indicates a lowering of the As–O bond order in the complexes which is supported by infrared measurements of the As–O stretching frequency (850 cm⁻¹ in the title complex relative to 880 cm⁻¹ in the free ligand).

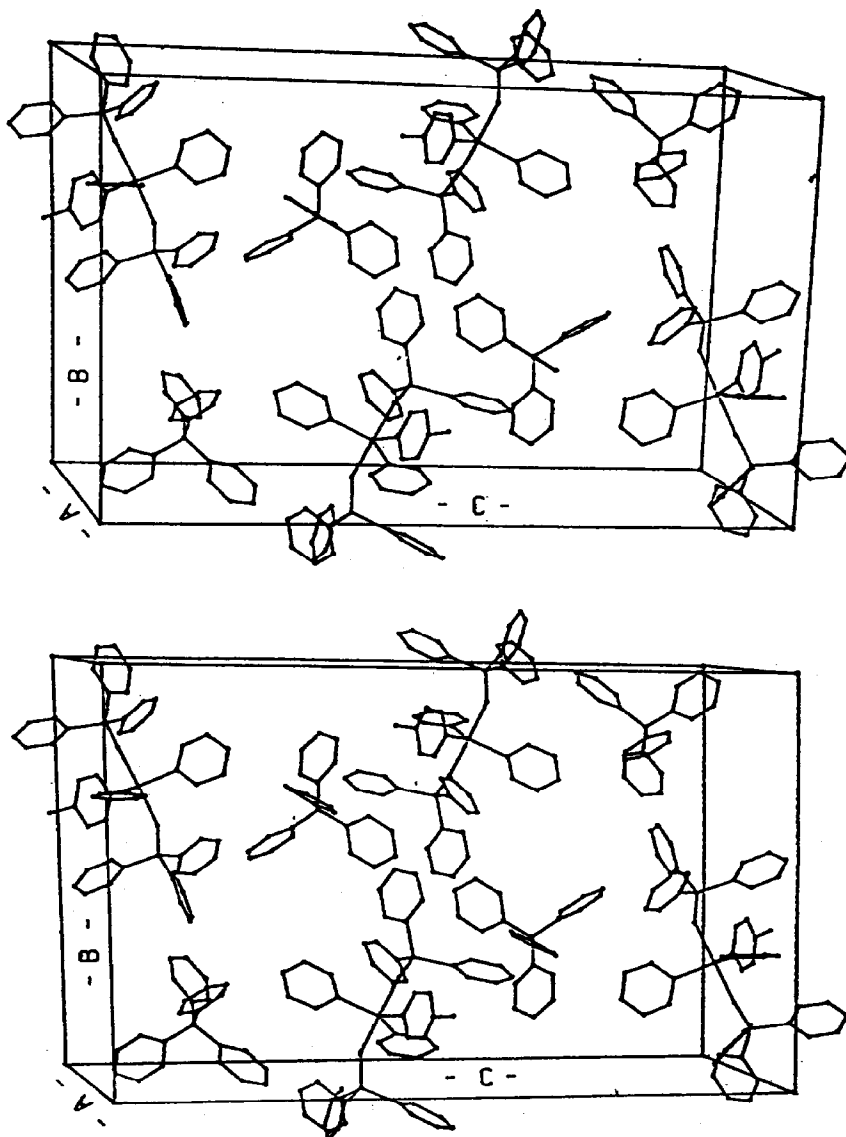


Fig. 2. Stereoscopic view of unit cell down the a axis.

The Sn–O bond lengths (2.205(5) and 2.219(5) Å) in **1** are comparable to that in $\text{Ph}_3\text{SnNO}_3 \cdot \text{Ph}_3\text{AsO}$ (2.181 Å [15]). By contrast, the Sn–O bonds involving coordinated hexamethylphosphoramide in the cationic complex, $[\text{Me}_3\text{Sn}(\text{HMPA})_2]^+ [\text{Me}_3\text{SnBr}_2]^-$ (2.12 Å [19]) are marginally shorter than in the corresponding neutral adduct, $\text{Me}_3\text{SnCl} \cdot \text{HMPA}$ [2.28 Å [19]), reflecting the enhanced acceptor strength of the triorganotin cation. In consequence, the Sn–O–P bond angle ($171(2)^\circ$) is seen to be widened in the cationic complex relative to the adduct ($158(1)^\circ$). A closer tin-oxygenated ligand interaction is also seen in the diaquotributyltin cation, $[\text{n-Bu}_3\text{Sn}(\text{H}_2\text{O})_2]^+ [\text{C}_5\text{CO}_2\text{Me}]^-$ (2.295, 2.326 Å [21]) compared to that in, for example, $\text{Me}_3\text{SnNO}_3 \cdot \text{H}_2\text{O}$ (2.47 Å [22]). It may be inferred from the foregoing that steric reasons preclude a discriminative interaction of triphenylarsine oxide with neutral

and cationic triaryltin acceptors in trigonal bipyramidal coordination. A closer approach of the ligand to the metal centre is, however, sterically permitted in the equatorial plane of the pentagonal bipyramidal structure of $\text{Ph}_2\text{Sn}(\text{NO}_3)_2 \cdot \text{Ph}_3\text{AsO}$ (Sn–O 2.02 Å [18]).

The geometry at the As atom in **1** is essentially tetrahedral, with the average (O–As–C) and (C–As–C) bond angles being $110.3(3)^\circ$ and $108.6(4)^\circ$, respectively; the corresponding values reported for the adduct, $\text{Ph}_3\text{SnNO}_3 \cdot \text{Ph}_3\text{AsO}$ are, respectively, 109.5° and 109.4° [15].

The geometry of the tetraphenylborate anion is also unequivocally tetrahedral. The average boron–ring carbon distance is 1.63(1) Å, and the average (C–B–C) bond angle is $109.5(8)^\circ$.

The intermolecular distances within the unit cell are normal. The closest contacts (excluding hydrogens) are between the C(54) atoms of two adjacent cations (symmetry: x, y, z and $\bar{x}, \bar{y}, 1 - z$) at a value of 3.452 Å, and between the C(64) atom of the cation and C(316) of the anion (symmetry transformation: $x - 1, \frac{1}{2} - y, z - \frac{1}{2}$) at a value of 3.496 Å. The shortest Sn–B distance is 8.914 Å, between B and Sn', where the primed atom is at symmetry transformation ($x, \frac{1}{2} - y, \frac{1}{2} + z$).

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References

- 1 P.J. Smith, *J. Organomet. Chem. Libr.*, 12 (1981) 97.
- 2 R.C. Poller, *Rev. Silicon, Germanium, Tin, Lead Compounds*, 3 (1978) 243.
- 3 S.W. Ng, Chen Wei, V.G. Kumar Das and T.C.W. Mak, *J. Organomet. Chem.*, 326 (1987) C61.
- 4 V.G. Kumar Das, W. Kitching and C.J. Moore, *J. Organomet. Chem.*, 22 (1970) 399.
- 5 V.G. Kumar Das, *J. Inorg. Nucl. Chem.*, 38 (1976) 1241.
- 6 G.M. Bancroft, V.G. Kumar Das, T.K. Sham and M.G. Clark, *J. Chem. Soc., Dalton Trans.*, (1979) 643.
- 7 R.R. Schrock and J.A. Osborn, *Inorg. Chem.*, 9 (1970) 2339.
- 8 M.J. Nolte, G. Gafner and L.M. Haines, *J. Chem. Soc., Chem. Commun.*, (1969) 1406.
- 9 C.B. Storm, C.M. Freeman, R.J. Butcher, A.H. Turner, N.S. Rowan, F.O. Johnson and E. Sinn, *Inorg. Chem.*, 22 (1983) 678.
- 10 TEXRAY System 234 Crystallographic Computing System, Molecular Structure Corporation, College Station, Texas 77840.
- 11 D.T. Cromer and J.T. Waber, *International Tables for X-ray Crystallography*, Kynoch Press, Birmingham, England, 1974; Vol. IV.
- 12 R.F. Stewart, E.R. Davidson and W.T. Simpson, *J. Chem. Phys.*, 42 (1965) 3175.
- 13 M. Nardelli, C. Pellizzi and G. Pellizzi, *J. Organomet. Chem.*, 112 (1976) 263.
- 14 V.G. Kumar Das, Chen Wei, S.W. Ng and T.C.W. Mak, *J. Organomet. Chem.*, 322 (1987) 33; V.G. Kumar Das, K.M. Lo, Chen Wei, S.J. Blunden and T.C.W. Mak, *ibid.*, 322 (1987) 163.
- 15 M. Nardelli, C. Pellizzi and G. Pellizzi, *J. Organomet. Chem.*, 125 (1977) 161.
- 16 C. Pellizzi and G. Pellizzi, *J. Chem. Soc., Dalton Trans.*, (1983) 847.
- 17 C. Dondi, M. Nardelli, C. Pellizzi, G. Pellizzi and G. Predieri, *J. Organomet. Chem.*, 308 (1986) 195.
- 18 M. Nardelli, C. Pellizzi, G. Pellizzi and P. Tarasconi, *Inorg. Chim. Acta*, 30 (1978) 179.

- 19 L.A. Aslanov, V.M. Attiya, V.M. Ionov, A.B. Permin and V.S. Petrosyan, *J. Struct. Chem. (Engl. Transl.)*, 18 (1977) 884.
- 20 G. Ferguson and E.W. Macaulay, *J. Chem. Soc., A*, (1969) 1.
- 21 A.G. Davies, J.P. Goddard, M.B. Hursthouse and N.P.C. Walker, *J. Chem. Soc., Chem. Commun.*, (1983) 597; *J. Chem. Soc., Dalton Trans.*, (1986) 1873.
- 22 R.E. Drew and F.W.B. Einstein, *Acta Crystallogr. B*, 28 (1972) 345.