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Bis(triphenylstannyl)borate toluene solvate

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The title compound, $[Sn_2(C_6H_5)_6(BHO_3)]\cdot C_7H_8$ or $(Ph_3SnO)_2B(OH)\cdot C_7H_8$, is an unexpected diester produced during an attempted recrystallization of $(Ph_3Sn)_3B_3O_3$. A planar central B atom is coordinated to two Ph_3Sn groups through ester linkages, and to a hydroxide group.

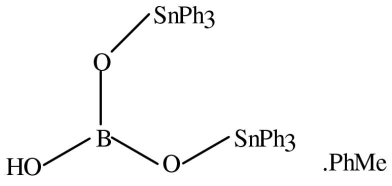
Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.009 Å
H-atom completeness 80%
Disorder in solvent or counterion
R factor = 0.033
wR factor = 0.078
Data-to-parameter ratio = 11.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The molecular structure obtained in this study (Fig. 1) is that of the toluene solvate of the bis(triphenylstannyl) ester of boric acid, $(Ph_3SnO)_2B(OH)\cdot C_7H_8$, (I), as opposed to the expected product, $(Ph_3Sn)_3B_3O_3$. Presumably it was formed as a hydrolysis product over the crystallization time period as a result of adventitious water in the (supposedly dried) solvents. Selected bond lengths and angles are given in Table 1. Few compounds containing the Sn—O—B link have been structurally characterized; these include $(Ph_3SnO)_3B$ (Ferguson *et al.*, 1995) $^{t}Bu_2Sn\{OB(OH)Ph\}_2$ and $^{t}Bu_2Sn(OH)_2\{(^{t}Bu_2SnO)_2OB(2,4,6\text{–}Me_3C_6H_2)\}_2\cdot 2MeCN$ (Brown *et al.*, 1992).



(I)

The core structure of (I) can be described as a planar three-coordinate sp^2-hybridized B atom bound to three O atoms with O—B—O angles of 118.3 (4)–121.7 (5)° (sum = 359.9°) and B—O distances of 1.353 (6)–1.392 (6) Å (average 1.369 Å). Of the three O atoms, O1 and O2 are two-coordinate and are respectively bound to Sn1 and Sn2, while O3 is presumably also two-coordinate and bound to an H atom. The B1—O3 bond distance [1.361 (6) Å] is not significantly different from the other two, and is close to their average (1.376 Å). The B—O distances and O—B—O angles are not significantly different from those reported for the tris(triphenylstannyl)borate ester (Ferguson *et al.*, 1995). The B—O—Sn angles, at 116.2 and 122.0°, indicate that the O atoms are sp^2-hybridized, but there has been considerable discussion over the remarkable flexibility of B—O—*M* (*M* = Sn, Ge, Si) bond angles in related compounds; these range from 112 to 140° (for Sn) (Ferguson *et al.*, 1995; Brown *et al.*, 1992), from 129 to 161° (for Si), and from 130 to 149° (for Ge) (Murphy *et al.*, 1993). The Sn atoms are each further bound to three phenyl groups with Sn—C distances of 2.119 (5)–2.139 (5) Å,

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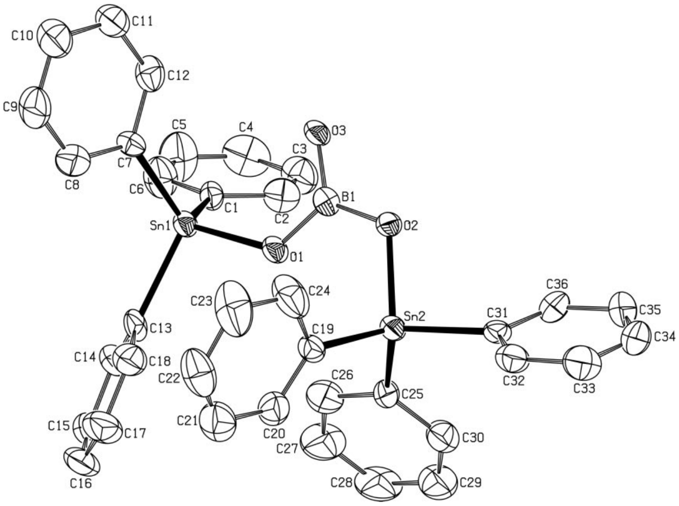

Figure 1
View of (I) (50% probability displacement ellipsoids).

(average 2.129 Å). The Sn atoms are not coplanar with the BO_3 moiety and the average Sn—O distance is 2.013 Å. The Sn atoms are four-coordinate and approximately tetrahedral, with O—Sn—C angles averaging 105.1° for Sn1 and 106.2° for Sn2, with C—Sn—C angles of 110.2 (2)–114.4 (2)°; the O1—Sn1—C13 and O2—Sn2—C31 angles, at 97.6 (2) and 99.3 (2)°, respectively, are considerably smaller than expected.

The crystal structure is composed of head-to-tail dimers, formed by interaction of a hydroxide with an ester oxygen [O3—H3···O2^i: O3···O2^i = 2.719 (4) Å; symmetry code: (i) 1−*x*, *y*, 1/2−*z*].

Experimental

An attempted recrystallization of $(Ph_3Sn)_3B_3O_3$ (Beckett *et al.*, 1999) from a solution of C_7H_8 layered with petroleum ether (313–333 K) yielded, after several weeks, crystalline material from which a few single crystals (m.p. 380–383 K) suitable for an X-ray structure determination were obtained.

Crystal data

$[Sn_2(C_6H_5)_6(BHO_3)]\cdot C_7H_8$
$M_r = 851.93$
Monoclinic, $C2/c$
$a = 18.344$ (3) Å
$b = 19.129$ (6) Å
$c = 22.252$ (5) Å
$\beta = 100.50$ (3)°
$V = 7678$ (3) Å^3
$Z = 8$

$D_x = 1.474$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 250 reflections
$\theta = 1.9$–24.8°
$\mu = 1.34$ mm^{-1}
$T = 150$ (2) K
Parallelepiped, colourless
0.22 × 0.20 × 0.08 mm

Data collection

Nonius FAST TV area-detector diffractometer
φ and ω scans (Darr *et al.*, 1993)
Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)
$T_{min} = 0.795$, $T_{max} = 0.900$
14 161 measured reflections

5411 independent reflections
3152 reflections with $I > 2\sigma(I)$
$R_{int} = 0.061$
$\theta_{max} = 24.8°$
$h = -20 \rightarrow 21$
$k = -20 \rightarrow 22$
$l = -16 \rightarrow 26$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.078$
$S = 0.88$
5411 reflections
481 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.029P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.87$ e Å^{-3}
$\Delta\rho_{min} = -0.50$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—O1	1.999 (3)	Sn2—C31	2.136 (4)
Sn1—C1	2.122 (5)	Sn2—C25	2.139 (5)
Sn1—C7	2.123 (5)	O1—B1	1.353 (6)
Sn1—C13	2.137 (5)	O2—B1	1.392 (6)
Sn2—O2	2.027 (3)	O3—B1	1.361 (6)
Sn2—C19	2.119 (5)		
O1—Sn1—C1	107.75 (17)	O2—Sn2—C25	110.86 (18)
O1—Sn1—C7	109.91 (18)	C19—Sn2—C25	113.1 (2)
C1—Sn1—C7	114.4 (2)	C31—Sn2—C25	110.2 (2)
O1—Sn1—C13	97.64 (15)	B1—O1—Sn1	122.0 (3)
C1—Sn1—C13	112.1 (2)	B1—O2—Sn2	116.2 (3)
C7—Sn1—C13	113.6 (2)	O1—B1—O3	120.0 (4)
O2—Sn2—C19	108.36 (19)	O1—B1—O2	118.3 (4)
O2—Sn2—C31	99.33 (15)	O3—B1—O2	121.7 (5)
C19—Sn2—C31	114.2 (2)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O2^i	0.82	1.91	2.719 (4)	172

Symmetry code: (i) $1 - x, y, \frac{1}{2} - z$.

The structure was found to contain partially occupied and disordered solvent which was modelled as toluene molecules to give best refinement results. The H atoms of the solvent were omitted, but those of the molecule of (I) were included in calculated positions using a riding model. All non-H atoms were refined anisotropically but it was necessary to restrain the C atoms of the toluene to approximate isotropic behaviour.

Data collection: *MADNES* (Pflugrath & Messerschmidt, 1989); cell refinement: *REFINE* in *MADNES*; data reduction: *MADNES*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1990); software used to prepare material for publication: *SHELXL*97.

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References

Beckett, M. A., Owen, P. & Varma, K. S. (1999). *J. Organomet. Chem.* **588**, 107–112.
Brown, P., Mahon, M. F. & Molloy, K. C. (1992). *J. Chem. Soc. Dalton Trans.* pp. 3503–3509.
Darr, J. A., Drake, S. R., Hursthouse, M. B. & Malik, K. M. A. (1993). *Inorg. Chem.* **32**, 5704–5708.
Ferguson, G., Spalding, T. R. & O'Dowd, A. T. (1995). *Acta Cryst.* C**51**, 67–70.

Murphy, D., Sheehan, J. P., Spalding, T. R., Ferguson, G., Lough, A. J. & Gallagher, J. F. (1993). *J. Mater. Chem.* **3**, 1275–1283.

Pflugrath, J. W. & Messerschmidt, A. (1989). *MADNES*. Version of 11th September. Delft Instruments, Delft, The Netherlands.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.

Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.