# Crystal structure of a 1,1-dibutyl-1H,3H-naphtho[1,8-cd][1,2,6]oxastannaborinin-3-ol



**Author(s):**
Breitwieser, Kevin; Chen, Peter

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# Crystal structure of a 1,1-dibutyl-1*H*,3*H*-naphtho-[1,8-*cd*][1,2,6]oxastannaborinin-3-ol

**Kevin Breitwieser and Peter Chen***

Laboratorium für Organische Chemie, ETH Zürich, Zürich, Switzerland. *Correspondence e-mail: peter.chen@org.chem.ethz.ch

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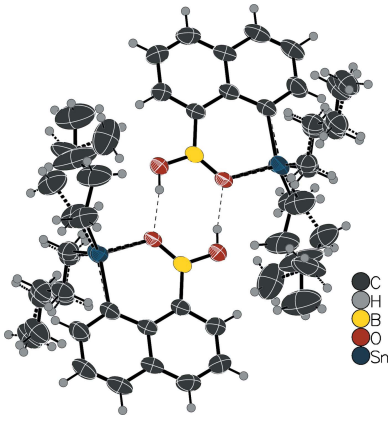
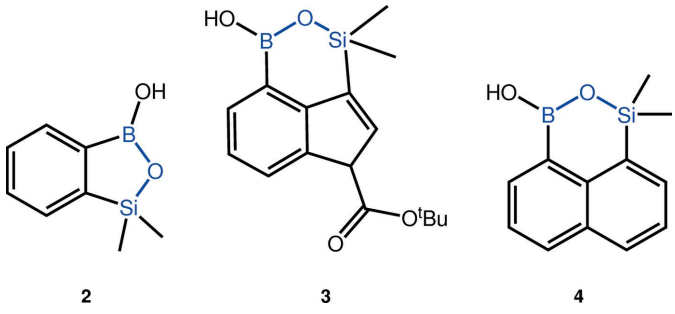
The title oxastannaborininol compound, $[Sn(C_4H_9)_2(C_{10}H_7BO_2)]$, has been synthesized and crystallized. While heterocycles containing a C–O–B group are common, heterocycles containing an *E*–O–B unit, where *E* is an element of the carbon group except for carbon, are rare. In fact, while heterocycles containing Si–O–B units are occasionally reported (although without crystal structures), there are no reports for the corresponding germanium, tin or lead analogues. Herein, the first synthesis and crystal structure of a heterocycle containing an Sn–O–B unit is described. The asymmetric unit contains one molecule showing a notable disorder of the tin atom and the butyl groups. They occupy two sets of positions with site-occupancy factors of 0.295 (6) and 0.705 (6).

## 1. Chemical context

Both tin and boron organic compounds are widespread reagents for cross-coupling reactions in organic synthesis (Negishi, 2002). The combination of tin- and boron-containing groups in one molecule can be advantageous, as they can undergo cross-coupling under different conditions. While the stannyl group easily undergoes transmetalation at elevated temperatures, a boronic acid will not do so with an additional activator, usually a base (Cárdenas, 2003). However, those groups are not usually connected. The only reported use of esters of stannanols and boronic acids lies in their increased Lewis acidity compared to the free boronic acid (Beckett *et al.*, 1999). They have been otherwise mentioned only in one publication, although no applications were reported (Murphy *et al.*, 1993).

Heterocycles containing an *E*–O–B unit (*E* = Si, Ge, Sn, Pb) have so far only been reported for silicon (Fig. 1). Benzosiloxaboroles, containing a five-membered ring with an Si–O–B



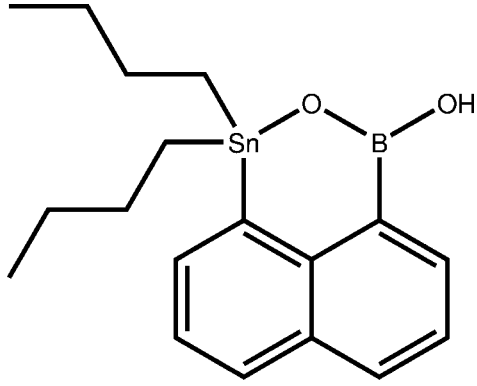




2 3 4

**Figure 1**
Chemical structure of compounds known in the literature that contain a heterocycle with an Si–O–B unit. Compound **2** (Brzozowska *et al.*, 2015) is a benzosiloxaborole, while compound **3** (Sumida *et al.*, 2018) and compound **4** (Su *et al.*, 2018) are oxasilaborininols.

unit, have shown promising properties for medical applications, being strong antimicrobial (Durka *et al.*, 2019) and antifungal agents (Brzozowska *et al.*, 2015).

Oxasilaboroninols have only been described in two cases. Sumida and co-workers accidentally stumbled upon **3** while trying to synthesize an oxasilole. They showed that both organometallic moieties can be replaced successively through Suzuki–Miyaura and Hiyama coupling (Sumida *et al.*, 2018). Su and Hartwig on the other hand synthesized oxasiliaboroninol **4** using ruthenium catalysis (Su *et al.*, 2018). In their report, they describe multiple transformations for this product, being able to replace selectively the boronic acid group while leaving a silanol group behind.




## 2. Structural commentary

The title molecule (**1**) is a cyclic intramolecular ester of a boronic acid and a stannanol. The asymmetric unit contains one molecule (Fig. 2). It shows notable disorder of the tin atom and the butyl groups. They occupy two sets of positions with site-occupancy factors of 0.295 (6) and 0.705 (6). It is furthermore planar, pointing towards electron delocalization over almost the whole molecule. The C—C bond lengths in the naphthalene structure are between 1.352 (4) and 1.439 (3) Å. This is in line with the bond lengths in naphthalene ranging from 1.350 to 1.421 Å (Abrahams *et al.*, 1949). The Sn—O bond distance is 2.0041 (17) and 2.040 (3) Å and the Sn—C bond connecting the tin atom to the aromatic ring has a length of 2.151 (6) Å and 2.210 (4) Å, varying due to disorder. The B—C bond has a length of 1.594 (3) Å, the B—O bond lengths are 1.352 (3) Å (B—OSn) and 1.362 (3) Å (B—OH).

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O2—H2···O1$^{i}$ | 0.88 (2) | 1.93 (2) | 2.805 (2) | 172 (3) |

Symmetry code: (i) $-x+\frac{3}{2}, -y+\frac{3}{2}, -z$.

## 3. Supramolecular features

In the crystal, the molecules form dimers through pairs of hydrogen bonds between the ring oxygen atom and the hydroxyl group with a distance of 2.805 (2) Å between the two involved oxygen atoms (Fig. 3, Table 1).

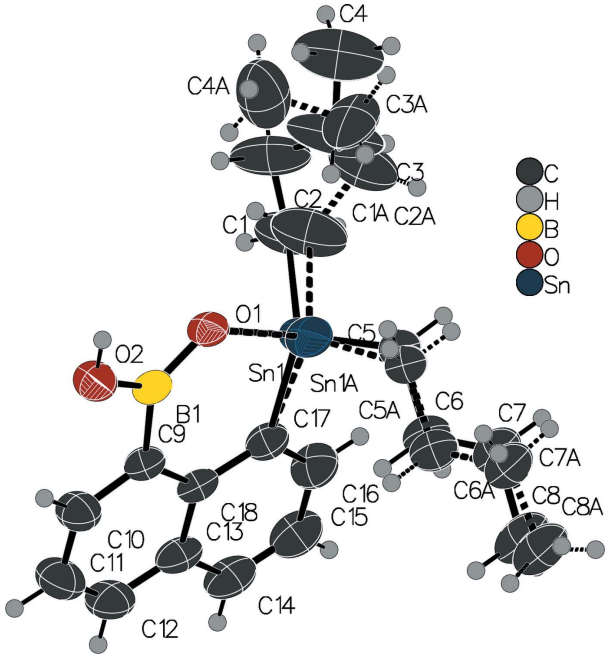



**Figure 2**
Crystal structure of the title compound **1**. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen atoms are drawn as fixed-size spheres with a radius of 0.15 Å. The tin atom and the butyl groups show notable disorder.

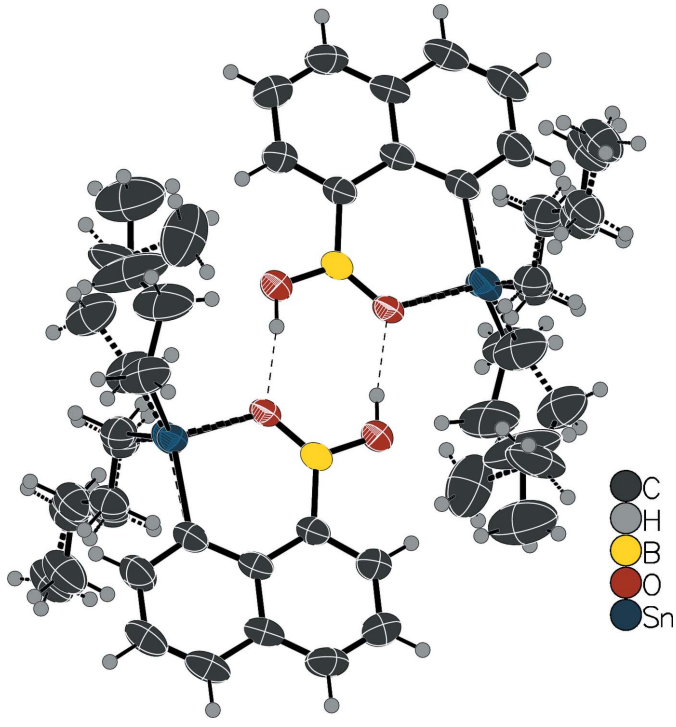



**Figure 3**
Structure of the dimer formed through hydrogen bonding. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen atoms are drawn as fixed-size spheres with a radius of 0.15 Å.

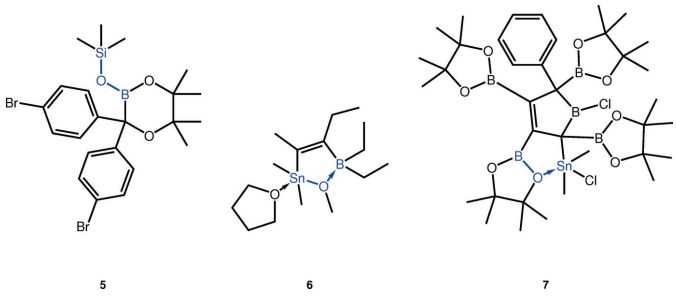



**Figure 4**
Structures of the compounds with the C–B–O–*E*–C motif (*E* = Si, Ge, Sn, Pb) and reported crystal structures. Compound **5** is the only one where this motif is formed by covalent bonds only (Ito *et al.*, 2011), while the compounds **6** (Boese *et al.*, 1996) and **7** (Braunschweig *et al.*, 2017) contain coordinative bonds.

## 4. Database survey

Searching the Cambridge Structural Database (CSD, version 5.41, update of November 2019; Groom *et al.*, 2016), not a single ester of a boronic acid and a stannanol has been crystallized. The same is true for the corresponding germanol and plumbanol derivatives. One ester of a boronic acid and trimethylsilanol (**5**) has been crystallized (Ito *et al.*, 2011). Two additional crystal structures containing the C–B–O–Sn–C motif have been reported. However, in those cases, either the O—Sn bond in **6** (Braunschweig *et al.*, 2017) or the O—B bond in **7** (Boese *et al.*, 1996) are not covalent, but rather coordinative bonds. Those three molecules are shown in Fig. 4.

The CSD lists three stannanols, all of which are triaryl stannanols (Růžička *et al.*, 2013; Barbul *et al.*, 2012). For those compounds, the Sn—O bond has a length of 1.981 to 2.057 Å, agreeing with the bond length of 2.0041 (17) Å found for the title compound. The Sn—$C_{Ar}$ bond length varies between 2.143 and 2.208 Å, matching the corresponding bond in the title compound.

## 5. Synthesis and crystallization

8-Iodo-1-naphthylboronic acid was prepared according to literature (Katz, 1986). Under argon, 122.6 mg (0.412 mmol, 1 eq.) of 8-iodo-1-naphthylboronic acid and 0.13 mL (0.459 mmol, 1.1 eq.) of tributyltin methoxide were heated to 373 K for 22.5 h; 0.2 mL (0.706 mmol, 1.7 eq.) of tributyltin methoxide were added and stirring was continued for 21 h at 373 K. Then 0.5 mL (1.764 mmol, 4.3 eq.) of tributyltin methoxide were added and the mixture was heated to 403 K for an additional 23 h. The mixture was cooled to RT and diluted by the addition of hexane. It was washed with equal volume 1 *M* aq. NaOH, dried ($Na_2SO_4$), filtered and concentrated *in vacuo*. The residue was purified by column chromatography (pure hexane to hexane:ethyl acetate 1:1) to obtain a yellowish solid that was crystallized by slow evaporation of a solution in 1,2-dimethoxyethane at 258 K and washed with pentane to obtain 27.3 mg (0.068 mmol, 15%) of colorless crystals suitable for X-ray crystallography.

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $[Sn(C_4H_9)_2(C_{10}H_7BO_2)]$ |
| $M_r$ | 402.88 |
| Crystal system, space group | Monoclinic, *C*2/*c* |
| Temperature (K) | 200 |
| *a*, *b*, *c* (Å) | 30.1386 (6), 11.2948 (1), 16.4726 (3) |
| $\beta$ (°) | 139.457 (4) |
| $V$ (Å$^3$) | 3644.9 (2) |
| $Z$ | 8 |
| Radiation type | Cu $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 11.17 |
| Crystal size (mm) | 0.26 × 0.13 × 0.02 |
| | |
| Data collection | |
| Diffractometer | Rigaku Oxford Diffraction XtaLAB Synergy, Dualflex, Pilatus 300K |
| Absorption correction | Gaussian (*CrysAlis PRO*; Rigaku OD, 2018) |
| $T_{min}$, $T_{max}$ | 0.234, 1.000 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 28847, 3923, 3724 |
| $R_{int}$ | 0.033 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.638 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.024, 0.066, 1.08 |
| No. of reflections | 3923 |
| No. of parameters | 264 |
| No. of restraints | 167 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.45, −0.39 |

Computer programs: *CrysAlis PRO* (Rigaku OD, 2018), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

$^1$H NMR (400 MHz, $CDCl_3$) $\delta$ 8.44 (*dd*, *J* = 7.0, 1.5 Hz, 1H, H12), 7.93 (*dd*, *J* = 8.2, 1.5 Hz, 1H, H10), 7.89 (*dd*, *J* = 7.1, 2.6 Hz, 1H, H14), 7.55 (*dd*, *J* = 8.1, 7.0 Hz, 1H, H11), 7.51–7.38 (*m*, 2H, H15 & H16), 4.80–4.37 (*s*, 1H, OH), 1.67 (*dtd*, *J* = 14.3, 7.2, 2.5 Hz, 4H, H2*A* & H2*B* & H6*A* & H6*B*), 1.47–1.29 (*m*, 8H H1*A* & H1*B* & H3*A* & H3*B* & H5*A* & H5*B* & H7*A* & H7*B*), 0.87 (*t*, *J* = 7.3 Hz, 6H H4*A* & H4*B* & H4*C* & H8*A* & H8*B* & H8*C*).

$^{11}$B NMR (128 MHz, $CDCl_3$) $\delta$ 27.22 (*s*, *br*, B1).

$^{13}$C NMR (101 MHz, $CDCl_3$) $\delta$ 142.65 (*s*, C18), 139.20 (*s*, C13), 137.30 (*s*, C12), 134.50 (*s*, C16), 133.78 (*s*, C17), 132.13 (*s*, C10), 130.81 (*s*, C14), 125.86 (*s*, C11), 124.55 (*s*, C15), 27.55 (*s*, C2 & C6), 27.10 (*s*, C3 & C7), 17.51 (*s*, C1 & C5), 13.70 (C4 & C8). C9 is not visible due to C–B interactions.

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Data were collected at 200 K, as a phase transition leads to breaking crystals at lower temperatures. The disordered tin atom and butyl groups were restrained using rigid body (RIGU) restraints with $\sigma$ for 1–3 distances and 1–2 distances of 0.004 and same-distance (SADI) restrains were applied to equivalent 1,2- and 1,3-distances within the disorder. Ellipsoids of four atoms and

their equivalents in the alternate orientation were constrained to be equal (EADP). H atoms were refined with riding coordinates [C—H = 0.93–0.97; $U_{iso}$(H) = 1.2$U_{eq}$(C) or 1.5$U_{eq}$(O, C-methyl)] except for the proton involved in the hydrogen bond, which was only lightly restrained with DFIX.

## Acknowledgements

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# supporting information

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# Crystal structure of a 1,1-dibutyl-1*H*,3*H*-naphtho[1,8-*cd*][1,2,6]oxastannaborinin-3-ol

**Kevin Breitwieser and Peter Chen**

## Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2018); cell refinement: *CrysAlis PRO* (Rigaku OD, 2018); data reduction: *CrysAlis PRO* (Rigaku OD, 2018); program(s) used to solve structure: ShelXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018/3* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

### 1,1-Dibutyl-1*H*,3*H*-naphtho[1,8-*cd*][1,2,6]oxastannaborinin-3-ol

*Crystal data*

$[Sn(C_4H_9)_2(C_{10}H_7BO_2)]$
$M_r$ = 402.88
Monoclinic, *C*2/*c*
*a* = 30.1386 (6) Å
*b* = 11.2948 (1) Å
*c* = 16.4726 (3) Å
*β* = 139.457 (4)°
*V* = 3644.9 (2) Å$^3$
*Z* = 8
*F*(000) = 1632
$D_x$ = 1.468 Mg m$^{-3}$
Cu *Kα* radiation, *λ* = 1.54184 Å
Cell parameters from 21527 reflections
*θ* = 4.5–79.4°
*μ* = 11.17 mm$^{-1}$
*T* = 200 K
Plate, clear colourless
0.26 × 0.13 × 0.02 mm

*Data collection*

Rigaku Oxford Diffraction XtaLAB Synergy, Dualflex, Pilatus 300K diffractometer
*ω* scans
Absorption correction: gaussian (CrysAlisPro; Rigaku OD, 2018)
$T_{min}$ = 0.234, $T_{max}$ = 1.000
28847 measured reflections
3923 independent reflections
3724 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.033
$\theta_{max}$ = 79.6°, $\theta_{min}$ = 4.5°
*h* = −38→29
*k* = −14→14
*l* = −17→20

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.066
*S* = 1.08
3923 reflections
264 parameters
167 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0359P)^2 + 2.7329P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.45 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.39 e Å$^{-3}$

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Sn1 | 0.80127 (4) | 0.57065 (8) | 0.24979 (9) | 0.03908 (16) | 0.705 (6) |
| O1 | 0.75421 (8) | 0.66412 (13) | 0.09740 (13) | 0.0452 (3) | |
| O2 | 0.66330 (9) | 0.77545 (16) | −0.07920 (16) | 0.0560 (4) | |
| H2 | 0.6923 (15) | 0.790 (3) | −0.079 (3) | 0.084* | |
| C18 | 0.65789 (11) | 0.61897 (17) | 0.12184 (19) | 0.0411 (4) | |
| C17 | 0.72252 (13) | 0.56497 (18) | 0.2265 (2) | 0.0465 (5) | |
| C9 | 0.64190 (11) | 0.68493 (18) | 0.02737 (19) | 0.0427 (4) | |
| C13 | 0.60618 (13) | 0.6048 (2) | 0.1109 (2) | 0.0498 (5) | |
| C16 | 0.73393 (15) | 0.5022 (2) | 0.3129 (3) | 0.0606 (6) | |
| H16 | 0.776391 | 0.467551 | 0.380439 | 0.073* | |
| C12 | 0.54116 (13) | 0.6536 (3) | 0.0087 (3) | 0.0628 (6) | |
| H12 | 0.507856 | 0.643613 | 0.002056 | 0.075* | |
| C15 | 0.68324 (17) | 0.4895 (3) | 0.3016 (3) | 0.0676 (7) | |
| H15 | 0.692174 | 0.447318 | 0.361363 | 0.081* | |
| B1 | 0.69046 (12) | 0.7075 (2) | 0.0188 (2) | 0.0403 (4) | |
| C14 | 0.62094 (16) | 0.5390 (2) | 0.2029 (3) | 0.0609 (6) | |
| H14 | 0.587231 | 0.529701 | 0.195310 | 0.073* | |
| C10 | 0.57682 (13) | 0.7313 (2) | −0.0696 (2) | 0.0571 (6) | |
| H10 | 0.565960 | 0.775310 | −0.130655 | 0.069* | |
| C11 | 0.52614 (14) | 0.7150 (3) | −0.0803 (3) | 0.0698 (7) | |
| H11 | 0.482665 | 0.746192 | −0.148228 | 0.084* | |
| C7 | 0.9197 (5) | 0.8346 (9) | 0.5547 (9) | 0.0641 (11) | 0.705 (6) |
| H7A | 0.927199 | 0.892715 | 0.522732 | 0.077* | 0.705 (6) |
| H7B | 0.962017 | 0.791753 | 0.621729 | 0.077* | 0.705 (6) |
| C6 | 0.8653 (4) | 0.7496 (5) | 0.4523 (7) | 0.0553 (11) | 0.705 (6) |
| H6A | 0.823204 | 0.793127 | 0.385509 | 0.066* | 0.705 (6) |
| H6B | 0.857484 | 0.692717 | 0.484475 | 0.066* | 0.705 (6) |
| C1 | 0.8221 (5) | 0.3983 (7) | 0.2290 (11) | 0.0757 (19) | 0.705 (6) |
| H1A | 0.854211 | 0.357484 | 0.308673 | 0.091* | 0.705 (6) |
| H1B | 0.780202 | 0.352665 | 0.169737 | 0.091* | 0.705 (6) |
| C5 | 0.8825 (3) | 0.6821 (5) | 0.3978 (6) | 0.0555 (11) | 0.705 (6) |
| H5A | 0.923203 | 0.634959 | 0.462947 | 0.067* | 0.705 (6) |
| H5B | 0.891816 | 0.738315 | 0.367705 | 0.067* | 0.705 (6) |
| C8 | 0.9005 (7) | 0.8980 (10) | 0.6062 (8) | 0.0776 (15) | 0.705 (6) |
| H8A | 0.855991 | 0.932701 | 0.538591 | 0.116* | 0.705 (6) |
| H8B | 0.933280 | 0.959032 | 0.662845 | 0.116* | 0.705 (6) |
| H8C | 0.900010 | 0.842311 | 0.649544 | 0.116* | 0.705 (6) |
| C3A | 0.9362 (9) | 0.469 (2) | 0.2939 (13) | 0.131 (9) | 0.295 (6) |
| H3AA | 0.980912 | 0.440122 | 0.340706 | 0.158* | 0.295 (6) |

| | | | | | |
|---|---|---|---|---|---|
| H3AB | 0.940412 | 0.552561 | 0.313166 | 0.158* | 0.295 (6) |
| C2 | 0.8516 (4) | 0.4040 (4) | 0.1841 (8) | 0.110 (2) | 0.705 (6) |
| H2A | 0.859231 | 0.324071 | 0.175231 | 0.132* | 0.705 (6) |
| H2B | 0.818392 | 0.441013 | 0.102315 | 0.132* | 0.705 (6) |
| C4 | 0.9615 (5) | 0.4669 (7) | 0.2585 (10) | 0.142 (3) | 0.705 (6) |
| H4A | 0.943675 | 0.519424 | 0.193075 | 0.213* | 0.705 (6) |
| H4B | 0.962191 | 0.387520 | 0.238623 | 0.213* | 0.705 (6) |
| H4C | 1.007051 | 0.490615 | 0.335834 | 0.213* | 0.705 (6) |
| C4A | 0.8861 (8) | 0.4541 (14) | 0.1525 (11) | 0.117 (6) | 0.295 (6) |
| H4AA | 0.840486 | 0.445560 | 0.109470 | 0.176* | 0.295 (6) |
| H4AB | 0.898299 | 0.384852 | 0.138891 | 0.176* | 0.295 (6) |
| H4AC | 0.888484 | 0.522479 | 0.121278 | 0.176* | 0.295 (6) |
| C3 | 0.9174 (5) | 0.4718 (7) | 0.2707 (12) | 0.149 (5) | 0.705 (6) |
| H3A | 0.906412 | 0.554416 | 0.264650 | 0.179* | 0.705 (6) |
| H3B | 0.945236 | 0.446432 | 0.354910 | 0.179* | 0.705 (6) |
| C2A | 0.9121 (6) | 0.4051 (10) | 0.3302 (14) | 0.091 (4) | 0.295 (6) |
| H2AA | 0.941594 | 0.426381 | 0.416263 | 0.110* | 0.295 (6) |
| H2AB | 0.919817 | 0.321779 | 0.330202 | 0.110* | 0.295 (6) |
| C1A | 0.8408 (11) | 0.4164 (14) | 0.261 (3) | 0.100 (8) | 0.295 (6) |
| H1AA | 0.833837 | 0.357857 | 0.293132 | 0.120* | 0.295 (6) |
| H1AB | 0.810547 | 0.396319 | 0.173984 | 0.120* | 0.295 (6) |
| Sn1A | 0.8101 (2) | 0.5864 (4) | 0.2649 (3) | 0.0696 (7) | 0.295 (6) |
| C5A | 0.8830 (9) | 0.7164 (16) | 0.4067 (17) | 0.0555 (11) | 0.295 (6) |
| H5AA | 0.926737 | 0.677223 | 0.474024 | 0.067* | 0.295 (6) |
| H5AB | 0.888251 | 0.774727 | 0.371250 | 0.067* | 0.295 (6) |
| C6A | 0.8671 (11) | 0.7814 (15) | 0.4627 (19) | 0.0553 (11) | 0.295 (6) |
| H6AA | 0.827565 | 0.831359 | 0.398553 | 0.066* | 0.295 (6) |
| H6AB | 0.854627 | 0.723469 | 0.486213 | 0.066* | 0.295 (6) |
| C7A | 0.9231 (13) | 0.856 (3) | 0.574 (2) | 0.0641 (11) | 0.295 (6) |
| H7AA | 0.931733 | 0.920516 | 0.547875 | 0.077* | 0.295 (6) |
| H7AB | 0.964334 | 0.808857 | 0.633969 | 0.077* | 0.295 (6) |
| C8A | 0.9093 (18) | 0.908 (3) | 0.637 (2) | 0.0776 (15) | 0.295 (6) |
| H8AA | 0.879434 | 0.974810 | 0.589615 | 0.116* | 0.295 (6) |
| H8AB | 0.951534 | 0.932796 | 0.719433 | 0.116* | 0.295 (6) |
| H8AC | 0.888151 | 0.849241 | 0.640933 | 0.116* | 0.295 (6) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Sn1 | 0.0426 (2) | 0.0423 (2) | 0.04336 (19) | 0.00587 (15) | 0.03572 (17) | 0.00775 (15) |
| O1 | 0.0546 (8) | 0.0530 (8) | 0.0488 (7) | 0.0110 (6) | 0.0450 (7) | 0.0129 (6) |
| O2 | 0.0562 (9) | 0.0733 (11) | 0.0550 (9) | 0.0151 (8) | 0.0468 (8) | 0.0221 (8) |
| C18 | 0.0557 (11) | 0.0377 (9) | 0.0490 (10) | −0.0046 (8) | 0.0451 (10) | −0.0059 (8) |
| C17 | 0.0602 (13) | 0.0475 (11) | 0.0514 (12) | −0.0012 (9) | 0.0478 (11) | 0.0012 (8) |
| C9 | 0.0521 (11) | 0.0445 (10) | 0.0475 (10) | 0.0001 (8) | 0.0423 (10) | −0.0010 (8) |
| C13 | 0.0656 (13) | 0.0488 (11) | 0.0632 (13) | −0.0119 (10) | 0.0568 (12) | −0.0116 (10) |
| C16 | 0.0771 (16) | 0.0629 (15) | 0.0624 (14) | 0.0033 (12) | 0.0587 (14) | 0.0109 (11) |
| C12 | 0.0598 (14) | 0.0779 (17) | 0.0756 (16) | −0.0075 (12) | 0.0584 (14) | −0.0072 (13) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C15 | 0.095 (2) | 0.0702 (16) | 0.0759 (17) | −0.0076 (15) | 0.0759 (17) | 0.0055 (13) |
| B1 | 0.0510 (12) | 0.0406 (10) | 0.0437 (11) | 0.0022 (9) | 0.0399 (10) | 0.0014 (9) |
| C14 | 0.0834 (17) | 0.0627 (14) | 0.0767 (16) | −0.0206 (13) | 0.0720 (16) | −0.0132 (13) |
| C10 | 0.0570 (13) | 0.0690 (15) | 0.0602 (13) | 0.0105 (11) | 0.0486 (12) | 0.0125 (11) |
| C11 | 0.0553 (14) | 0.091 (2) | 0.0741 (17) | 0.0102 (13) | 0.0522 (14) | 0.0107 (15) |
| C7 | 0.084 (2) | 0.058 (4) | 0.062 (3) | 0.003 (2) | 0.059 (2) | −0.0001 (19) |
| C6 | 0.0703 (16) | 0.047 (4) | 0.066 (2) | −0.001 (3) | 0.0567 (16) | −0.002 (2) |
| C1 | 0.093 (4) | 0.040 (2) | 0.132 (5) | 0.010 (2) | 0.096 (4) | 0.012 (3) |
| C5 | 0.0517 (13) | 0.066 (4) | 0.0520 (16) | 0.003 (2) | 0.0403 (13) | −0.006 (2) |
| C8 | 0.123 (4) | 0.062 (3) | 0.078 (5) | 0.003 (3) | 0.084 (5) | 0.000 (3) |
| C3A | 0.094 (10) | 0.25 (3) | 0.098 (9) | −0.067 (13) | 0.087 (8) | −0.059 (12) |
| C2 | 0.163 (6) | 0.063 (3) | 0.188 (7) | 0.031 (3) | 0.157 (6) | 0.013 (3) |
| C4 | 0.176 (8) | 0.098 (4) | 0.244 (11) | 0.024 (5) | 0.185 (9) | 0.019 (6) |
| C4A | 0.095 (10) | 0.087 (9) | 0.094 (8) | 0.003 (7) | 0.050 (8) | −0.011 (7) |
| C3 | 0.165 (7) | 0.094 (5) | 0.291 (13) | 0.022 (5) | 0.202 (9) | 0.007 (6) |
| C2A | 0.095 (8) | 0.061 (6) | 0.146 (12) | 0.022 (5) | 0.099 (9) | 0.031 (7) |
| C1A | 0.124 (13) | 0.057 (9) | 0.17 (2) | 0.030 (9) | 0.127 (16) | 0.039 (10) |
| Sn1A | 0.0836 (12) | 0.0915 (15) | 0.0617 (9) | 0.0361 (8) | 0.0630 (9) | 0.0338 (8) |
| C5A | 0.0517 (13) | 0.066 (4) | 0.0520 (16) | 0.003 (2) | 0.0403 (13) | −0.006 (2) |
| C6A | 0.0703 (16) | 0.047 (4) | 0.066 (2) | −0.001 (3) | 0.0567 (16) | −0.002 (2) |
| C7A | 0.084 (2) | 0.058 (4) | 0.062 (3) | 0.003 (2) | 0.059 (2) | −0.0001 (19) |
| C8A | 0.123 (4) | 0.062 (3) | 0.078 (5) | 0.003 (3) | 0.084 (5) | 0.000 (3) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Sn1—O1 | 2.0041 (17) | C8—H8A | 0.9600 |
| Sn1—C17 | 2.098 (3) | C8—H8B | 0.9600 |
| Sn1—C1 | 2.151 (6) | C8—H8C | 0.9600 |
| Sn1—C5 | 2.110 (5) | C3A—H3AA | 0.9700 |
| O1—B1 | 1.352 (3) | C3A—H3AB | 0.9700 |
| O1—Sn1A | 2.040 (3) | C3A—C4A | 1.546 (13) |
| O2—H2 | 0.882 (18) | C3A—C2A | 1.439 (12) |
| O2—B1 | 1.362 (3) | C2—H2A | 0.9700 |
| C18—C17 | 1.427 (3) | C2—H2B | 0.9700 |
| C18—C9 | 1.439 (3) | C2—C3 | 1.501 (9) |
| C18—C13 | 1.435 (3) | C4—H4A | 0.9600 |
| C17—C16 | 1.380 (3) | C4—H4B | 0.9600 |
| C17—Sn1A | 2.210 (4) | C4—H4C | 0.9600 |
| C9—B1 | 1.594 (3) | C4—C3 | 1.489 (8) |
| C9—C10 | 1.382 (3) | C4A—H4AA | 0.9600 |
| C13—C12 | 1.401 (4) | C4A—H4AB | 0.9600 |
| C13—C14 | 1.422 (4) | C4A—H4AC | 0.9600 |
| C16—H16 | 0.9300 | C3—H3A | 0.9700 |
| C16—C15 | 1.399 (4) | C3—H3B | 0.9700 |
| C12—H12 | 0.9300 | C2A—H2AA | 0.9700 |
| C12—C11 | 1.352 (4) | C2A—H2AB | 0.9700 |
| C15—H15 | 0.9300 | C2A—C1A | 1.483 (13) |
| C15—C14 | 1.357 (4) | C1A—H1AA | 0.9700 |

| | | | |
|---|---|---|---|
| C14—H14 | 0.9300 | C1A—H1AB | 0.9700 |
| C10—H10 | 0.9300 | C1A—Sn1A | 2.157 (13) |
| C10—C11 | 1.410 (3) | Sn1A—C5A | 2.154 (12) |
| C11—H11 | 0.9300 | C5A—H5AA | 0.9700 |
| C7—H7A | 0.9700 | C5A—H5AB | 0.9700 |
| C7—H7B | 0.9700 | C5A—C6A | 1.513 (12) |
| C7—C6 | 1.500 (6) | C6A—H6AA | 0.9700 |
| C7—C8 | 1.521 (7) | C6A—H6AB | 0.9700 |
| C6—H6A | 0.9700 | C6A—C7A | 1.489 (13) |
| C6—H6B | 0.9700 | C7A—H7AA | 0.9700 |
| C6—C5 | 1.535 (6) | C7A—H7AB | 0.9700 |
| C1—H1A | 0.9700 | C7A—C8A | 1.498 (13) |
| C1—H1B | 0.9700 | C8A—H8AA | 0.9600 |
| C1—C2 | 1.530 (9) | C8A—H8AB | 0.9600 |
| C5—H5A | 0.9700 | C8A—H8AC | 0.9600 |
| C5—H5B | 0.9700 | | |

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|---|---|---|---|
| O1—Sn1—C17 | 99.30 (8) | H8B—C8—H8C | 109.5 |
| O1—Sn1—C1 | 107.0 (3) | H3AA—C3A—H3AB | 108.1 |
| O1—Sn1—C5 | 103.2 (2) | C4A—C3A—H3AA | 109.6 |
| C17—Sn1—C1 | 111.7 (2) | C4A—C3A—H3AB | 109.6 |
| C17—Sn1—C5 | 112.8 (2) | C2A—C3A—H3AA | 109.6 |
| C5—Sn1—C1 | 120.1 (3) | C2A—C3A—H3AB | 109.6 |
| B1—O1—Sn1 | 121.92 (13) | C2A—C3A—C4A | 110.4 (11) |
| B1—O1—Sn1A | 124.28 (15) | C1—C2—H2A | 108.9 |
| B1—O2—H2 | 113 (2) | C1—C2—H2B | 108.9 |
| C17—C18—C9 | 123.78 (18) | H2A—C2—H2B | 107.7 |
| C17—C18—C13 | 117.34 (19) | C3—C2—C1 | 113.2 (7) |
| C13—C18—C9 | 118.9 (2) | C3—C2—H2A | 108.9 |
| C18—C17—Sn1 | 121.60 (15) | C3—C2—H2B | 108.9 |
| C18—C17—Sn1A | 122.42 (16) | H4A—C4—H4B | 109.5 |
| C16—C17—Sn1 | 117.84 (19) | H4A—C4—H4C | 109.5 |
| C16—C17—C18 | 120.5 (2) | H4B—C4—H4C | 109.5 |
| C16—C17—Sn1A | 117.0 (2) | C3—C4—H4A | 109.5 |
| C18—C9—B1 | 127.12 (19) | C3—C4—H4B | 109.5 |
| C10—C9—C18 | 117.46 (19) | C3—C4—H4C | 109.5 |
| C10—C9—B1 | 115.39 (18) | C3A—C4A—H4AA | 109.5 |
| C12—C13—C18 | 120.0 (2) | C3A—C4A—H4AB | 109.5 |
| C12—C13—C14 | 120.3 (2) | C3A—C4A—H4AC | 109.5 |
| C14—C13—C18 | 119.6 (2) | H4AA—C4A—H4AB | 109.5 |
| C17—C16—H16 | 119.2 | H4AA—C4A—H4AC | 109.5 |
| C17—C16—C15 | 121.7 (3) | H4AB—C4A—H4AC | 109.5 |
| C15—C16—H16 | 119.2 | C2—C3—H3A | 107.0 |
| C13—C12—H12 | 119.5 | C2—C3—H3B | 106.9 |
| C11—C12—C13 | 121.0 (2) | C4—C3—C2 | 121.5 (9) |
| C11—C12—H12 | 119.5 | C4—C3—H3A | 106.9 |
| C16—C15—H15 | 120.2 | C4—C3—H3B | 106.9 |
| C14—C15—C16 | 119.6 (2) | H3A—C3—H3B | 106.7 |

| | | | |
|---|---|---|---|
| C14—C15—H15 | 120.2 | C3A—C2A—H2AA | 106.9 |
| O1—B1—O2 | 118.44 (18) | C3A—C2A—H2AB | 106.9 |
| O1—B1—C9 | 126.12 (18) | C3A—C2A—C1A | 121.8 (14) |
| O2—B1—C9 | 115.44 (18) | H2AA—C2A—H2AB | 106.7 |
| C13—C14—H14 | 119.4 | C1A—C2A—H2AA | 106.9 |
| C15—C14—C13 | 121.3 (2) | C1A—C2A—H2AB | 106.9 |
| C15—C14—H14 | 119.4 | C2A—C1A—H1AA | 108.0 |
| C9—C10—H10 | 118.5 | C2A—C1A—H1AB | 108.0 |
| C9—C10—C11 | 123.1 (2) | C2A—C1A—Sn1A | 117.2 (11) |
| C11—C10—H10 | 118.5 | H1AA—C1A—H1AB | 107.2 |
| C12—C11—C10 | 119.6 (3) | Sn1A—C1A—H1AA | 108.0 |
| C12—C11—H11 | 120.2 | Sn1A—C1A—H1AB | 108.0 |
| C10—C11—H11 | 120.2 | O1—Sn1A—C17 | 94.66 (15) |
| H7A—C7—H7B | 107.9 | O1—Sn1A—C1A | 105.8 (10) |
| C6—C7—H7A | 109.2 | O1—Sn1A—C5A | 106.7 (7) |
| C6—C7—H7B | 109.2 | C1A—Sn1A—C17 | 110.1 (4) |
| C6—C7—C8 | 112.1 (6) | C5A—Sn1A—C17 | 113.4 (5) |
| C8—C7—H7A | 109.2 | C5A—Sn1A—C1A | 122.2 (8) |
| C8—C7—H7B | 109.2 | Sn1A—C5A—H5AA | 108.0 |
| C7—C6—H6A | 108.7 | Sn1A—C5A—H5AB | 108.0 |
| C7—C6—H6B | 108.7 | H5AA—C5A—H5AB | 107.3 |
| C7—C6—C5 | 114.2 (5) | C6A—C5A—Sn1A | 117.2 (11) |
| H6A—C6—H6B | 107.6 | C6A—C5A—H5AA | 108.0 |
| C5—C6—H6A | 108.7 | C6A—C5A—H5AB | 108.0 |
| C5—C6—H6B | 108.7 | C5A—C6A—H6AA | 108.3 |
| Sn1—C1—H1A | 109.1 | C5A—C6A—H6AB | 108.3 |
| Sn1—C1—H1B | 109.1 | H6AA—C6A—H6AB | 107.4 |
| H1A—C1—H1B | 107.8 | C7A—C6A—C5A | 115.7 (13) |
| C2—C1—Sn1 | 112.7 (5) | C7A—C6A—H6AA | 108.3 |
| C2—C1—H1A | 109.1 | C7A—C6A—H6AB | 108.3 |
| C2—C1—H1B | 109.1 | C6A—C7A—H7AA | 108.6 |
| Sn1—C5—H5A | 109.4 | C6A—C7A—H7AB | 108.6 |
| Sn1—C5—H5B | 109.4 | C6A—C7A—C8A | 114.7 (14) |
| C6—C5—Sn1 | 111.2 (4) | H7AA—C7A—H7AB | 107.6 |
| C6—C5—H5A | 109.4 | C8A—C7A—H7AA | 108.6 |
| C6—C5—H5B | 109.4 | C8A—C7A—H7AB | 108.6 |
| H5A—C5—H5B | 108.0 | C7A—C8A—H8AA | 109.5 |
| C7—C8—H8A | 109.5 | C7A—C8A—H8AB | 109.5 |
| C7—C8—H8B | 109.5 | C7A—C8A—H8AC | 109.5 |
| C7—C8—H8C | 109.5 | H8AA—C8A—H8AB | 109.5 |
| H8A—C8—H8B | 109.5 | H8AA—C8A—H8AC | 109.5 |
| H8A—C8—H8C | 109.5 | H8AB—C8A—H8AC | 109.5 |

| | | | |
|---|---|---|---|
| Sn1—O1—B1—O2 | 176.60 (15) | C13—C18—C17—C16 | −0.2 (3) |
| Sn1—O1—B1—C9 | −4.7 (3) | C13—C18—C17—Sn1A | −176.2 (2) |
| Sn1—C17—C16—C15 | −177.2 (2) | C13—C18—C9—B1 | −177.74 (19) |
| Sn1—C1—C2—C3 | 59.5 (9) | C13—C18—C9—C10 | 0.0 (3) |
| C18—C17—C16—C15 | −0.1 (4) | C13—C12—C11—C10 | −0.7 (5) |

| | | | |
|---|---|---|---|
| C18—C9—B1—O1 | 2.6 (3) | C16—C15—C14—C13 | −0.6 (4) |
| C18—C9—B1—O2 | −178.7 (2) | C12—C13—C14—C15 | 179.0 (3) |
| C18—C9—C10—C11 | −1.1 (4) | B1—C9—C10—C11 | 176.9 (3) |
| C18—C13—C12—C11 | −0.4 (4) | C14—C13—C12—C11 | −179.1 (3) |
| C18—C13—C14—C15 | 0.3 (4) | C10—C9—B1—O1 | −175.2 (2) |
| C17—C18—C9—B1 | 1.6 (3) | C10—C9—B1—O2 | 3.6 (3) |
| C17—C18—C9—C10 | 179.3 (2) | C7—C6—C5—Sn1 | −177.6 (7) |
| C17—C18—C13—C12 | −178.6 (2) | C1—C2—C3—C4 | 167.2 (8) |
| C17—C18—C13—C14 | 0.1 (3) | C8—C7—C6—C5 | −179.4 (8) |
| C17—C16—C15—C14 | 0.5 (4) | C3A—C2A—C1A—Sn1A | −66 (3) |
| C9—C18—C17—Sn1 | −2.6 (3) | C4A—C3A—C2A—C1A | −49 (3) |
| C9—C18—C17—C16 | −179.5 (2) | Sn1A—O1—B1—O2 | 168.6 (2) |
| C9—C18—C17—Sn1A | 4.4 (3) | Sn1A—O1—B1—C9 | −12.7 (3) |
| C9—C18—C13—C12 | 0.8 (3) | Sn1A—C17—C16—C15 | 176.1 (3) |
| C9—C18—C13—C14 | 179.5 (2) | Sn1A—C5A—C6A—C7A | 170.7 (19) |
| C9—C10—C11—C12 | 1.5 (5) | C5A—C6A—C7A—C8A | −173 (2) |
| C13—C18—C17—Sn1 | 176.78 (15) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O2—H2···O1$^{i}$ | 0.88 (2) | 1.93 (2) | 2.805 (2) | 172 (3) |

Symmetry code: (i) −*x*+3/2, −*y*+3/2, −*z*.