

# Triclinic modification of di-*n*-butylbis(2-hydroxybenzoato- $\kappa^2O^1,O^1$ )tin(IV)

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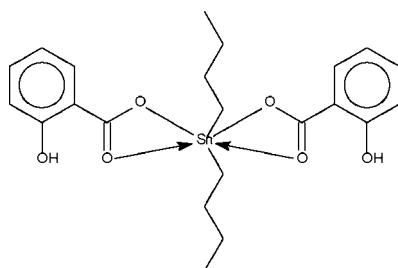
Received 22 July 2008; accepted 28 July 2008

Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.014$  Å;  $R$  factor = 0.060;  $wR$  factor = 0.189; data-to-parameter ratio = 19.3.

The Sn atom in the title compound,  $[\text{Sn}(\text{C}_4\text{H}_9)_2(\text{C}_7\text{H}_5\text{O}_3)_2]$ , is chelated by the carboxylate groups of 2-hydroxybenzoate ligands, and exists in a six-coordinate skew-trapezoidal bipyramidal coordination geometry [ $\text{C}-\text{Sn}-\text{C} = 140.1$  ( $3^\circ$ )].

## Related literature

For the monoclinic modification, see: Narula *et al.* (1992). For a review of the structural chemistry of organotin carboxylates, see: Tiekink (1991, 1994). For a discussion of skew-trapezoidal bipyramidal diorganotin bis(chelates), see: Ng *et al.* (1987).



## Experimental

### Crystal data

$[\text{Sn}(\text{C}_4\text{H}_9)_2(\text{C}_7\text{H}_5\text{O}_3)_2]$   
 $M_r = 507.13$   
Triclinic,  $P\bar{1}$   
 $a = 9.1652$  (2) Å  
 $b = 11.2111$  (2) Å

$c = 12.2620$  (2) Å  
 $\alpha = 94.759$  ( $1^\circ$ )  
 $\beta = 106.872$  ( $1^\circ$ )  
 $\gamma = 108.586$  ( $1^\circ$ )  
 $V = 1121.24$  (4) Å<sup>3</sup>

$Z = 2$   
Mo  $K\alpha$  radiation  
 $\mu = 1.17$  mm<sup>-1</sup>

$T = 100$  (2) K  
 $0.25 \times 0.20 \times 0.15$  mm

### Data collection

Bruker SMART APEX  
diffractometer  
Absorption correction: multi-scan  
(*SADABS*; Sheldrick, 1996)  
 $T_{\min} = 0.758$ ,  $T_{\max} = 0.844$

11666 measured reflections  
5068 independent reflections  
4633 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.034$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.059$   
 $wR(F^2) = 0.189$   
 $S = 1.18$   
5068 reflections  
262 parameters

2 restraints  
H-atom parameters constrained  
 $\Delta\rho_{\max} = 2.57$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -1.40$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$                         | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---------------------------------------|-------|-------------|-------------|---------------|
| $\text{O3}-\text{H3o}\cdots\text{O2}$ | 0.84  | 1.96        | 2.599 (9)   | 132           |
| $\text{O6}-\text{H6o}\cdots\text{O5}$ | 0.84  | 2.00        | 2.626 (8)   | 131           |

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINTE* (Bruker, 2007); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2286).

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## supporting information

*Acta Cryst.* (2008). E64, m1187 [doi:10.1107/S1600536808023799]

**Triclinic modification of di-*n*-butylbis(2-hydroxybenzoato- $\kappa^2O^1, O^1'$ )tin(IV)**

Reza Reisi, Misni Misran, Kong Mun Lo and Seik Weng Ng

**S1. Comment**

Diorganotin dicarboxylates generally exist as monomeric molecules in which the carboxylate groups chelate in an anisobidentate manner (Tiekink, 1991; 1994). The  $R_2Sn$  unit is bent, and the geometry at tin is described as being skew-trapezoidal bipyramidal (Ng *et al.*, 1987). The title compound has been reported in a monoclinic form (Narula *et al.*, 1992). This structure has one *n*-butyl group in a *W* conformation and the other in a *U* conformation. In the present triclinic modification (Scheme I, Fig. 1), both groups adopt a *W* conformation. Intramolecular O-H $\cdots$ O hydrogen bonds are noted (Table 1).

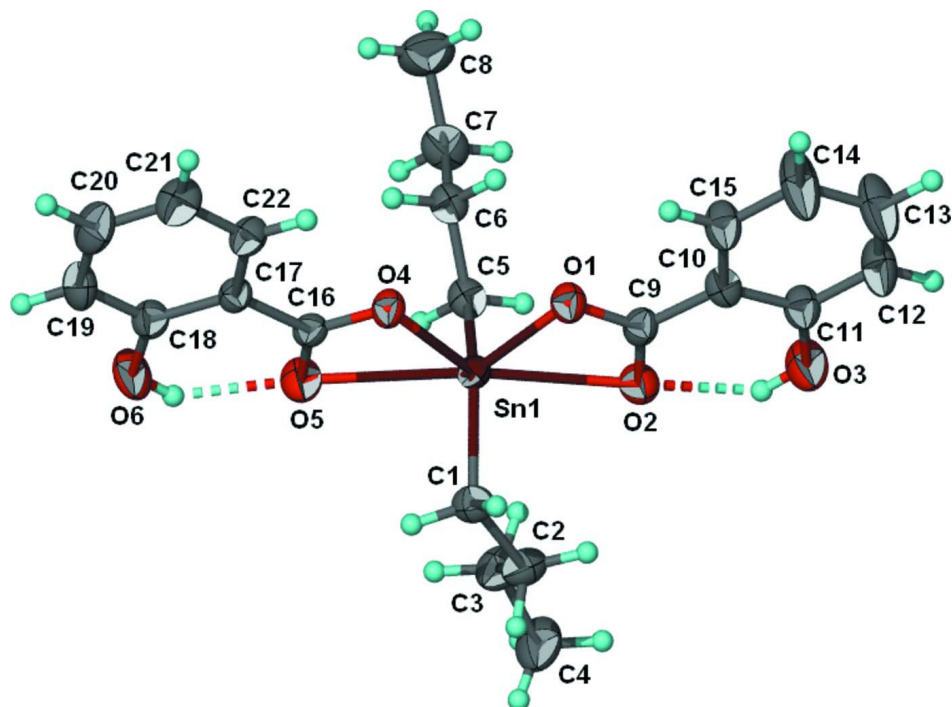
**S2. Experimental**

Diethyltin oxide (2 g, 8 mmol) and salicylic acid (2.2 g, 16 mmol) were heated in toluene (100 ml) in a Dean-Stark water apparatus. Slow evaporation of the filtered solution yielded colorless crystals.

**S3. Refinement**

Carbon-bound H-atoms were placed in positions (C–H 0.95 to 0.99 Å) and were included in the refinement in the riding model approximation, with  $U(H)$  set to 1.2–1.5 $U_{eq}(C)$ . The hydroxy H-atoms were similarly constrained (O–H 0.84 Å) but the hybridization of the oxygen atoms was assumed to be  $sp^2$ .

The final difference Fourier map had a peak of 2.57 e Å<sup>-3</sup> at 1.5 Å from the O5 and O6 atoms, and a deep hole of -1.40 e Å<sup>-3</sup> at 1.5 Å from the H12 atom.

**Figure 1**

Thermal ellipsoid plot (Barbour, 2001) plot of the triclinic form of  $[\text{Sn}(\text{C}_4\text{H}_9)_2(\text{C}_7\text{H}_5\text{O}_3)]$  at the 50% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

### di-*n*-butylbis(2-hydroxybenzoato- $\kappa^2\text{O}^1, \text{O}^1$ )tin(IV)

#### Crystal data

$[\text{Sn}(\text{C}_4\text{H}_9)_2(\text{C}_7\text{H}_5\text{O}_3)_2]$

$M_r = 507.13$

Triclinic,  $P\bar{1}$

Hall symbol: -P 1

$a = 9.1652(2) \text{ \AA}$

$b = 11.2111(2) \text{ \AA}$

$c = 12.2620(2) \text{ \AA}$

$\alpha = 94.759(1)^\circ$

$\beta = 106.872(1)^\circ$

$\gamma = 108.586(1)^\circ$

$V = 1121.24(4) \text{ \AA}^3$

$Z = 2$

$F(000) = 516$

$D_x = 1.502 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 6302 reflections

$\theta = 2.5\text{--}27.7^\circ$

$\mu = 1.17 \text{ mm}^{-1}$

$T = 100 \text{ K}$

Block, colorless

$0.25 \times 0.20 \times 0.15 \text{ mm}$

#### Data collection

Bruker SMART APEX

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega$  scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.758$ ,  $T_{\max} = 0.844$

11666 measured reflections

5068 independent reflections

4633 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.034$

$\theta_{\max} = 27.5^\circ$ ,  $\theta_{\min} = 1.8^\circ$

$h = -11 \rightarrow 7$

$k = -14 \rightarrow 14$

$l = -15 \rightarrow 15$

Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.059$

$wR(F^2) = 0.189$

$S = 1.18$

5068 reflections

262 parameters

2 restraints

Primary atom site location: structure-invariant  
direct methods

Secondary atom site location: difference Fourier  
map

Hydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0604P)^2 + 8.6498P]$

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

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 2.57 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -1.40 \text{ e } \text{\AA}^{-3}$

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

|     | x            | y           | z           | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|-------------|-------------|----------------------------------|
| Sn1 | 0.50157 (6)  | 0.34322 (5) | 0.64132 (4) | 0.03511 (17)                     |
| O1  | 0.6039 (7)   | 0.5445 (5)  | 0.6922 (5)  | 0.0424 (11)                      |
| O2  | 0.4303 (7)   | 0.4748 (5)  | 0.7850 (5)  | 0.0495 (13)                      |
| O3  | 0.4148 (9)   | 0.6313 (7)  | 0.9462 (6)  | 0.0687 (19)                      |
| H3O | 0.3802       | 0.5549      | 0.9111      | 0.103*                           |
| O4  | 0.6470 (6)   | 0.3786 (5)  | 0.5355 (4)  | 0.0391 (11)                      |
| O5  | 0.5384 (8)   | 0.1687 (6)  | 0.5037 (5)  | 0.0535 (14)                      |
| O6  | 0.5995 (9)   | 0.0298 (5)  | 0.3515 (6)  | 0.0634 (18)                      |
| H6O | 0.5475       | 0.0276      | 0.3980      | 0.095*                           |
| C1  | 0.2603 (9)   | 0.2912 (8)  | 0.5242 (7)  | 0.0452 (17)                      |
| H1A | 0.2522       | 0.3652      | 0.4873      | 0.054*                           |
| H1B | 0.2412       | 0.2201      | 0.4620      | 0.054*                           |
| C2  | 0.1264 (10)  | 0.2505 (8)  | 0.5755 (8)  | 0.0512 (19)                      |
| H2A | 0.1354       | 0.3253      | 0.6296      | 0.061*                           |
| H2B | 0.0198       | 0.2243      | 0.5123      | 0.061*                           |
| C3  | 0.1281 (12)  | 0.1422 (9)  | 0.6398 (9)  | 0.059 (2)                        |
| H3A | 0.2243       | 0.1738      | 0.7118      | 0.071*                           |
| H3B | 0.1410       | 0.0738      | 0.5911      | 0.071*                           |
| C4  | -0.0260 (15) | 0.0841 (11) | 0.6719 (11) | 0.077 (3)                        |
| H4A | -0.0159      | 0.0150      | 0.7140      | 0.116*                           |
| H4B | -0.1217      | 0.0496      | 0.6009      | 0.116*                           |
| H4C | -0.0389      | 0.1507      | 0.7212      | 0.116*                           |
| C5  | 0.6353 (10)  | 0.2839 (7)  | 0.7842 (7)  | 0.0429 (16)                      |
| H5A | 0.6076       | 0.3066      | 0.8534      | 0.051*                           |
| H5B | 0.6034       | 0.1895      | 0.7669      | 0.051*                           |
| C6  | 0.8165 (10)  | 0.3455 (7)  | 0.8106 (7)  | 0.0423 (16)                      |
| H6A | 0.8473       | 0.4399      | 0.8259      | 0.051*                           |
| H6B | 0.8436       | 0.3215      | 0.7415      | 0.051*                           |
| C7  | 0.9178 (12)  | 0.3068 (9)  | 0.9151 (8)  | 0.059 (2)                        |
| H7A | 0.8917       | 0.3318      | 0.9845      | 0.070*                           |
| H7B | 0.8861       | 0.2124      | 0.9003      | 0.070*                           |
| C8  | 1.0967 (13)  | 0.3666 (13) | 0.9401 (10) | 0.079 (3)                        |
| H8A | 1.1547       | 0.3411      | 1.0093      | 0.118*                           |
| H8B | 1.1288       | 0.4602      | 0.9538      | 0.118*                           |

|     |             |             |             |             |
|-----|-------------|-------------|-------------|-------------|
| H8C | 1.1245      | 0.3381      | 0.8736      | 0.118*      |
| C9  | 0.5369 (9)  | 0.5664 (7)  | 0.7668 (6)  | 0.0391 (15) |
| C10 | 0.5867 (10) | 0.6976 (7)  | 0.8301 (6)  | 0.0391 (15) |
| C11 | 0.5246 (11) | 0.7235 (8)  | 0.9170 (7)  | 0.0466 (18) |
| C12 | 0.5768 (15) | 0.8474 (9)  | 0.9784 (8)  | 0.066 (3)   |
| H12 | 0.5344      | 0.8648      | 1.0375      | 0.079*      |
| C13 | 0.6896 (18) | 0.9447 (9)  | 0.9535 (11) | 0.085 (4)   |
| H13 | 0.7237      | 1.0298      | 0.9949      | 0.102*      |
| C14 | 0.7559 (19) | 0.9206 (9)  | 0.8676 (11) | 0.091 (5)   |
| H14 | 0.8361      | 0.9884      | 0.8522      | 0.109*      |
| C15 | 0.7029 (12) | 0.7971 (8)  | 0.8060 (8)  | 0.053 (2)   |
| H15 | 0.7457      | 0.7799      | 0.7471      | 0.064*      |
| C16 | 0.6292 (8)  | 0.2670 (7)  | 0.4830 (6)  | 0.0350 (14) |
| C17 | 0.7181 (8)  | 0.2616 (7)  | 0.4013 (6)  | 0.0344 (14) |
| C18 | 0.6975 (10) | 0.1431 (7)  | 0.3404 (7)  | 0.0420 (16) |
| C19 | 0.7814 (12) | 0.1408 (9)  | 0.2613 (7)  | 0.052 (2)   |
| H19 | 0.7649      | 0.0615      | 0.2163      | 0.063*      |
| C20 | 0.8863 (13) | 0.2521 (10) | 0.2488 (8)  | 0.061 (2)   |
| H20 | 0.9441      | 0.2489      | 0.1964      | 0.074*      |
| C21 | 0.9105 (12) | 0.3695 (9)  | 0.3108 (8)  | 0.056 (2)   |
| H21 | 0.9849      | 0.4463      | 0.3020      | 0.067*      |
| C22 | 0.8242 (9)  | 0.3729 (7)  | 0.3858 (7)  | 0.0419 (16) |
| H22 | 0.8380      | 0.4532      | 0.4276      | 0.050*      |

*Atomic displacement parameters (Å<sup>2</sup>)*

|     | $U^{11}$   | $U^{22}$   | $U^{33}$   | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|-----|------------|------------|------------|--------------|--------------|--------------|
| Sn1 | 0.0326 (3) | 0.0370 (3) | 0.0356 (3) | 0.00980 (19) | 0.01499 (19) | 0.00458 (18) |
| O1  | 0.048 (3)  | 0.040 (3)  | 0.042 (3)  | 0.015 (2)    | 0.020 (2)    | 0.006 (2)    |
| O2  | 0.049 (3)  | 0.045 (3)  | 0.052 (3)  | 0.010 (2)    | 0.021 (3)    | 0.006 (2)    |
| O3  | 0.078 (5)  | 0.059 (4)  | 0.071 (4)  | 0.009 (3)    | 0.048 (4)    | 0.003 (3)    |
| O4  | 0.044 (3)  | 0.038 (3)  | 0.041 (3)  | 0.014 (2)    | 0.022 (2)    | 0.006 (2)    |
| O5  | 0.058 (4)  | 0.046 (3)  | 0.060 (4)  | 0.010 (3)    | 0.035 (3)    | 0.010 (3)    |
| O6  | 0.076 (4)  | 0.036 (3)  | 0.076 (4)  | 0.006 (3)    | 0.041 (4)    | −0.002 (3)   |
| C1  | 0.041 (4)  | 0.054 (4)  | 0.039 (4)  | 0.014 (3)    | 0.013 (3)    | 0.011 (3)    |
| C2  | 0.040 (4)  | 0.051 (5)  | 0.065 (5)  | 0.017 (4)    | 0.020 (4)    | 0.019 (4)    |
| C3  | 0.055 (5)  | 0.055 (5)  | 0.077 (6)  | 0.019 (4)    | 0.034 (5)    | 0.022 (5)    |
| C4  | 0.080 (7)  | 0.070 (7)  | 0.098 (8)  | 0.019 (6)    | 0.062 (7)    | 0.014 (6)    |
| C5  | 0.050 (4)  | 0.040 (4)  | 0.039 (4)  | 0.012 (3)    | 0.019 (3)    | 0.010 (3)    |
| C6  | 0.048 (4)  | 0.039 (4)  | 0.041 (4)  | 0.013 (3)    | 0.017 (3)    | 0.010 (3)    |
| C7  | 0.063 (6)  | 0.057 (5)  | 0.054 (5)  | 0.024 (4)    | 0.011 (4)    | 0.016 (4)    |
| C8  | 0.056 (6)  | 0.104 (9)  | 0.074 (7)  | 0.037 (6)    | 0.008 (5)    | 0.020 (6)    |
| C9  | 0.042 (4)  | 0.046 (4)  | 0.034 (3)  | 0.021 (3)    | 0.014 (3)    | 0.008 (3)    |
| C10 | 0.049 (4)  | 0.036 (3)  | 0.032 (3)  | 0.017 (3)    | 0.011 (3)    | 0.009 (3)    |
| C11 | 0.057 (5)  | 0.044 (4)  | 0.044 (4)  | 0.017 (4)    | 0.024 (4)    | 0.008 (3)    |
| C12 | 0.099 (8)  | 0.055 (5)  | 0.054 (5)  | 0.027 (5)    | 0.043 (5)    | 0.003 (4)    |
| C13 | 0.136 (11) | 0.037 (5)  | 0.086 (8)  | 0.010 (6)    | 0.068 (8)    | −0.001 (5)   |
| C14 | 0.145 (12) | 0.038 (5)  | 0.100 (9)  | 0.007 (6)    | 0.089 (9)    | 0.002 (5)    |

|     |           |           |           |           |           |           |
|-----|-----------|-----------|-----------|-----------|-----------|-----------|
| C15 | 0.073 (6) | 0.044 (4) | 0.051 (5) | 0.017 (4) | 0.038 (4) | 0.009 (4) |
| C16 | 0.033 (3) | 0.040 (4) | 0.033 (3) | 0.013 (3) | 0.012 (3) | 0.007 (3) |
| C17 | 0.036 (3) | 0.041 (4) | 0.031 (3) | 0.017 (3) | 0.013 (3) | 0.008 (3) |
| C18 | 0.043 (4) | 0.039 (4) | 0.046 (4) | 0.013 (3) | 0.020 (3) | 0.008 (3) |
| C19 | 0.068 (6) | 0.055 (5) | 0.041 (4) | 0.026 (4) | 0.025 (4) | 0.003 (3) |
| C20 | 0.074 (6) | 0.082 (7) | 0.048 (5) | 0.039 (5) | 0.036 (5) | 0.018 (4) |
| C21 | 0.064 (6) | 0.059 (5) | 0.060 (5) | 0.023 (4) | 0.038 (5) | 0.028 (4) |
| C22 | 0.044 (4) | 0.043 (4) | 0.048 (4) | 0.020 (3) | 0.022 (3) | 0.016 (3) |

*Geometric parameters (Å, °)*

|           |            |            |            |
|-----------|------------|------------|------------|
| Sn1—C1    | 2.118 (8)  | C6—H6A     | 0.9900     |
| Sn1—C5    | 2.117 (8)  | C6—H6B     | 0.9900     |
| Sn1—O1    | 2.106 (5)  | C7—C8      | 1.485 (15) |
| Sn1—O2    | 2.561 (6)  | C7—H7A     | 0.9900     |
| Sn1—O4    | 2.090 (5)  | C7—H7B     | 0.9900     |
| Sn1—O5    | 2.645 (6)  | C8—H8A     | 0.9800     |
| O1—C9     | 1.288 (9)  | C8—H8B     | 0.9800     |
| O2—C9     | 1.259 (9)  | C8—H8C     | 0.9800     |
| O3—C11    | 1.348 (10) | C9—C10     | 1.467 (10) |
| O3—H3O    | 0.8400     | C10—C11    | 1.396 (11) |
| O4—C16    | 1.296 (8)  | C10—C15    | 1.395 (11) |
| O5—C16    | 1.247 (9)  | C11—C12    | 1.386 (12) |
| O6—C18    | 1.346 (9)  | C12—C13    | 1.368 (15) |
| O6—H6O    | 0.8400     | C12—H12    | 0.9500     |
| C1—C2     | 1.501 (11) | C13—C14    | 1.408 (15) |
| C1—H1A    | 0.9900     | C13—H13    | 0.9500     |
| C1—H1B    | 0.9900     | C14—C15    | 1.382 (12) |
| C2—C3     | 1.503 (12) | C14—H14    | 0.9500     |
| C2—H2A    | 0.9900     | C15—H15    | 0.9500     |
| C2—H2B    | 0.9900     | C16—C17    | 1.472 (9)  |
| C3—C4     | 1.534 (13) | C17—C22    | 1.383 (10) |
| C3—H3A    | 0.9900     | C17—C18    | 1.397 (10) |
| C3—H3B    | 0.9900     | C18—C19    | 1.405 (11) |
| C4—H4A    | 0.9800     | C19—C20    | 1.362 (14) |
| C4—H4B    | 0.9800     | C19—H19    | 0.9500     |
| C4—H4C    | 0.9800     | C20—C21    | 1.382 (14) |
| C5—C6     | 1.503 (11) | C20—H20    | 0.9500     |
| C5—H5A    | 0.9900     | C21—C22    | 1.382 (11) |
| C5—H5B    | 0.9900     | C21—H21    | 0.9500     |
| C6—C7     | 1.533 (11) | C22—H22    | 0.9500     |
| O4—Sn1—O1 | 82.3 (2)   | H6A—C6—H6B | 107.7      |
| O4—Sn1—C5 | 104.7 (3)  | C8—C7—C6   | 113.3 (8)  |
| O1—Sn1—C5 | 102.1 (3)  | C8—C7—H7A  | 108.9      |
| O4—Sn1—C1 | 104.1 (3)  | C6—C7—H7A  | 108.9      |
| O1—Sn1—C1 | 108.7 (3)  | C8—C7—H7B  | 108.9      |
| C1—Sn1—C5 | 140.1 (3)  | C6—C7—H7B  | 108.9      |

|            |             |             |           |
|------------|-------------|-------------|-----------|
| O4—Sn1—O2  | 137.44 (19) | H7A—C7—H7B  | 107.7     |
| O1—Sn1—O2  | 55.25 (19)  | C7—C8—H8A   | 109.5     |
| C5—Sn1—O2  | 88.1 (3)    | C7—C8—H8B   | 109.5     |
| C1—Sn1—O2  | 89.0 (3)    | H8A—C8—H8B  | 109.5     |
| O4—Sn1—O5  | 53.64 (18)  | C7—C8—H8C   | 109.5     |
| O1—Sn1—O5  | 135.77 (19) | H8A—C8—H8C  | 109.5     |
| C5—Sn1—O5  | 87.8 (3)    | H8B—C8—H8C  | 109.5     |
| C1—Sn1—O5  | 87.5 (3)    | O2—C9—O1    | 119.5 (7) |
| O2—Sn1—O5  | 168.92 (18) | O2—C9—C10   | 120.8 (7) |
| C9—O1—Sn1  | 102.7 (5)   | O1—C9—C10   | 119.6 (7) |
| C9—O2—Sn1  | 82.4 (4)    | C11—C10—C15 | 119.7 (7) |
| C11—O3—H3O | 120.0       | C11—C10—C9  | 121.1 (7) |
| C16—O4—Sn1 | 106.0 (4)   | C15—C10—C9  | 119.2 (7) |
| C16—O5—Sn1 | 81.3 (4)    | O3—C11—C12  | 117.1 (8) |
| C18—O6—H6O | 120.0       | O3—C11—C10  | 122.6 (7) |
| C2—C1—Sn1  | 116.0 (5)   | C12—C11—C10 | 120.3 (8) |
| C2—C1—H1A  | 108.3       | C13—C12—C11 | 119.7 (9) |
| Sn1—C1—H1A | 108.3       | C13—C12—H12 | 120.1     |
| C2—C1—H1B  | 108.3       | C11—C12—H12 | 120.1     |
| Sn1—C1—H1B | 108.3       | C12—C13—C14 | 121.0 (9) |
| H1A—C1—H1B | 107.4       | C12—C13—H13 | 119.5     |
| C3—C2—C1   | 114.3 (7)   | C14—C13—H13 | 119.5     |
| C3—C2—H2A  | 108.7       | C15—C14—C13 | 119.2 (9) |
| C1—C2—H2A  | 108.7       | C15—C14—H14 | 120.4     |
| C3—C2—H2B  | 108.7       | C13—C14—H14 | 120.4     |
| C1—C2—H2B  | 108.7       | C14—C15—C10 | 120.2 (8) |
| H2A—C2—H2B | 107.6       | C14—C15—H15 | 119.9     |
| C2—C3—C4   | 114.0 (9)   | C10—C15—H15 | 119.9     |
| C2—C3—H3A  | 108.8       | O5—C16—O4   | 119.1 (6) |
| C4—C3—H3A  | 108.8       | O5—C16—C17  | 122.5 (7) |
| C2—C3—H3B  | 108.8       | O4—C16—C17  | 118.4 (6) |
| C4—C3—H3B  | 108.8       | C22—C17—C18 | 119.7 (7) |
| H3A—C3—H3B | 107.7       | C22—C17—C16 | 120.5 (6) |
| C3—C4—H4A  | 109.5       | C18—C17—C16 | 119.9 (6) |
| C3—C4—H4B  | 109.5       | O6—C18—C19  | 117.5 (7) |
| H4A—C4—H4B | 109.5       | O6—C18—C17  | 123.8 (7) |
| C3—C4—H4C  | 109.5       | C19—C18—C17 | 118.7 (7) |
| H4A—C4—H4C | 109.5       | C20—C19—C18 | 120.2 (8) |
| H4B—C4—H4C | 109.5       | C20—C19—H19 | 119.9     |
| C6—C5—Sn1  | 111.8 (5)   | C18—C19—H19 | 119.9     |
| C6—C5—H5A  | 109.2       | C19—C20—C21 | 121.5 (8) |
| Sn1—C5—H5A | 109.2       | C19—C20—H20 | 119.2     |
| C6—C5—H5B  | 109.2       | C21—C20—H20 | 119.2     |
| Sn1—C5—H5B | 109.2       | C22—C21—C20 | 118.6 (8) |
| H5A—C5—H5B | 107.9       | C22—C21—H21 | 120.7     |
| C5—C6—C7   | 113.4 (7)   | C20—C21—H21 | 120.7     |
| C5—C6—H6A  | 108.9       | C17—C22—C21 | 121.3 (8) |
| C7—C6—H6A  | 108.9       | C17—C22—H22 | 119.4     |

|               |            |                 |             |
|---------------|------------|-----------------|-------------|
| C5—C6—H6B     | 108.9      | C21—C22—H22     | 119.4       |
| C7—C6—H6B     | 108.9      |                 |             |
| O4—Sn1—O1—C9  | 179.6 (5)  | Sn1—O1—C9—O2    | -4.1 (8)    |
| C5—Sn1—O1—C9  | -76.9 (5)  | Sn1—O1—C9—C10   | 175.5 (5)   |
| C1—Sn1—O1—C9  | 77.3 (5)   | O2—C9—C10—C11   | 3.9 (11)    |
| O2—Sn1—O1—C9  | 2.1 (4)    | O1—C9—C10—C11   | -175.7 (7)  |
| O5—Sn1—O1—C9  | -176.4 (4) | O2—C9—C10—C15   | -178.8 (8)  |
| O4—Sn1—O2—C9  | -5.8 (6)   | O1—C9—C10—C15   | 1.7 (11)    |
| O1—Sn1—O2—C9  | -2.1 (4)   | C15—C10—C11—O3  | -178.5 (9)  |
| C5—Sn1—O2—C9  | 104.0 (5)  | C9—C10—C11—O3   | -1.2 (13)   |
| C1—Sn1—O2—C9  | -115.8 (5) | C15—C10—C11—C12 | 0.7 (13)    |
| O5—Sn1—O2—C9  | 172.4 (9)  | C9—C10—C11—C12  | 178.0 (8)   |
| O1—Sn1—O4—C16 | 176.7 (5)  | O3—C11—C12—C13  | 179.2 (11)  |
| C5—Sn1—O4—C16 | 76.2 (5)   | C10—C11—C12—C13 | 0.0 (17)    |
| C1—Sn1—O4—C16 | -75.8 (5)  | C11—C12—C13—C14 | -1 (2)      |
| O2—Sn1—O4—C16 | 179.8 (4)  | C12—C13—C14—C15 | 1 (2)       |
| O5—Sn1—O4—C16 | 0.2 (4)    | C13—C14—C15—C10 | -1 (2)      |
| O4—Sn1—O5—C16 | -0.2 (4)   | C11—C10—C15—C14 | -0.2 (15)   |
| O1—Sn1—O5—C16 | -5.1 (6)   | C9—C10—C15—C14  | -177.6 (10) |
| C5—Sn1—O5—C16 | -110.3 (5) | Sn1—O5—C16—O4   | 0.3 (6)     |
| C1—Sn1—O5—C16 | 109.4 (5)  | Sn1—O5—C16—C17  | -179.9 (7)  |
| O2—Sn1—O5—C16 | -178.7 (9) | Sn1—O4—C16—O5   | -0.4 (8)    |
| O4—Sn1—C1—C2  | 175.7 (6)  | Sn1—O4—C16—C17  | 179.8 (5)   |
| O1—Sn1—C1—C2  | -97.9 (6)  | O5—C16—C17—C22  | -176.5 (7)  |
| C5—Sn1—C1—C2  | 40.6 (9)   | O4—C16—C17—C22  | 3.2 (10)    |
| O2—Sn1—C1—C2  | -45.3 (6)  | O5—C16—C17—C18  | 2.4 (11)    |
| O5—Sn1—C1—C2  | 124.2 (6)  | O4—C16—C17—C18  | -177.9 (7)  |
| Sn1—C1—C2—C3  | -55.4 (10) | C22—C17—C18—O6  | 178.7 (8)   |
| C1—C2—C3—C4   | -169.0 (9) | C16—C17—C18—O6  | -0.2 (12)   |
| O4—Sn1—C5—C6  | 30.8 (6)   | C22—C17—C18—C19 | -2.3 (11)   |
| O1—Sn1—C5—C6  | -54.3 (6)  | C16—C17—C18—C19 | 178.8 (7)   |
| C1—Sn1—C5—C6  | 165.7 (5)  | O6—C18—C19—C20  | -177.9 (9)  |
| O2—Sn1—C5—C6  | -108.1 (5) | C17—C18—C19—C20 | 3.0 (13)    |
| O5—Sn1—C5—C6  | 82.2 (5)   | C18—C19—C20—C21 | -1.5 (15)   |
| Sn1—C5—C6—C7  | 178.9 (6)  | C19—C20—C21—C22 | -0.7 (15)   |
| C5—C6—C7—C8   | 179.3 (9)  | C18—C17—C22—C21 | 0.1 (12)    |
| Sn1—O2—C9—O1  | 3.3 (6)    | C16—C17—C22—C21 | 179.0 (7)   |
| Sn1—O2—C9—C10 | -176.3 (7) | C20—C21—C22—C17 | 1.4 (13)    |

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ )

| $D-H\cdots A$      | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--------------------|-------|-------------|-------------|---------------|
| O3—H3o $\cdots$ O2 | 0.84  | 1.96        | 2.599 (9)   | 132           |
| O6—H6o $\cdots$ O5 | 0.84  | 2.00        | 2.626 (8)   | 131           |