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## Structure Reports

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catena-Poly[[dimethyltin(IV)]- $\mu$ -cis-cyclohexane-1,2-dicarboxylato]

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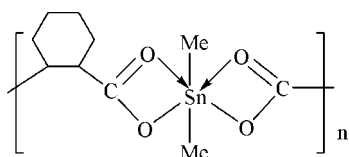
Received 3 January 2009; accepted 4 February 2009

Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å;  $R$  factor = 0.031;  $wR$  factor = 0.062; data-to-parameter ratio = 15.6.

The title complex,  $[\text{Sn}(\text{CH}_3)_2(\text{C}_8\text{H}_{10}\text{O}_4)]_n$ , was synthesized from *cis*-cyclohexane-1,2-dicarboxylic acid and dimethyltin dichloride. The complex has a bridging bis-bidentate carboxylate group resulting in a zig-zag chain structure parallel to [001]. The Sn atom is six-coordinated and displays a distorted octahedral geometry.

## Related literature

For background to organotin complexes, see: Gielen (2002); Han *et al.* (2007). For related structures, see: Swisher *et al.* (1984).



## Experimental

## Crystal data

$[\text{Sn}(\text{CH}_3)_2(\text{C}_8\text{H}_{10}\text{O}_4)]$   
 $M_r = 318.92$   
 Monoclinic,  $P2_1/c$

$a = 10.0880$  (16) Å  
 $b = 10.430$  (2) Å  
 $c = 11.592$  (2) Å

$\beta = 99.041$  (2)°  
 $V = 1204.5$  (4) Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation

$\mu = 2.11$  mm<sup>-1</sup>  
 $T = 298$  (2) K  
 $0.32 \times 0.19 \times 0.17$  mm

## Data collection

Bruker SMART CCD area-detector diffractometer  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.551$ ,  $T_{\max} = 0.715$

6188 measured reflections  
 2117 independent reflections  
 1822 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.022$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$   
 $wR(F^2) = 0.062$   
 $S = 1.19$   
 2117 reflections

136 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.52$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.43$  e Å<sup>-3</sup>

Table 1

Selected geometric parameters (Å, °).

|            |             |        |           |
|------------|-------------|--------|-----------|
| Sn1—O3     | 2.089 (3)   | Sn1—O1 | 2.102 (3) |
| Sn1—C9     | 2.089 (4)   | Sn1—O4 | 2.570 (3) |
| Sn1—C10    | 2.098 (4)   | Sn1—O2 | 2.660 (3) |
| C9—Sn1—C10 | 137.14 (18) |        |           |

Data collection: SMART (Bruker, 1996); cell refinement: SAINT (Bruker, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2192).

## References

- Bruker (1996). SMART and SAINT. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Gielen, M. (2002). *Appl. Organomet. Chem.* **16**, 481–494.  
 Han, Y., Zhang, R. & Wang, D. (2007). *Acta Cryst.* **E63**, m776–m777.  
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.  
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
 Swisher, R. G., Vollano, J. F., Chandrasekhar, V., Day, R. O. & Holmes, R. R. (1984). *Inorg. Chem.* **23**, 3147–3152.

## supporting information

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**catena-Poly[[dimethyltin(IV)]- $\mu$ -cis-cyclohexane-1,2-dicarboxylato]**

Yuerong Wang, Rufen Zhang and Yongxin Li

**S1. Comment**

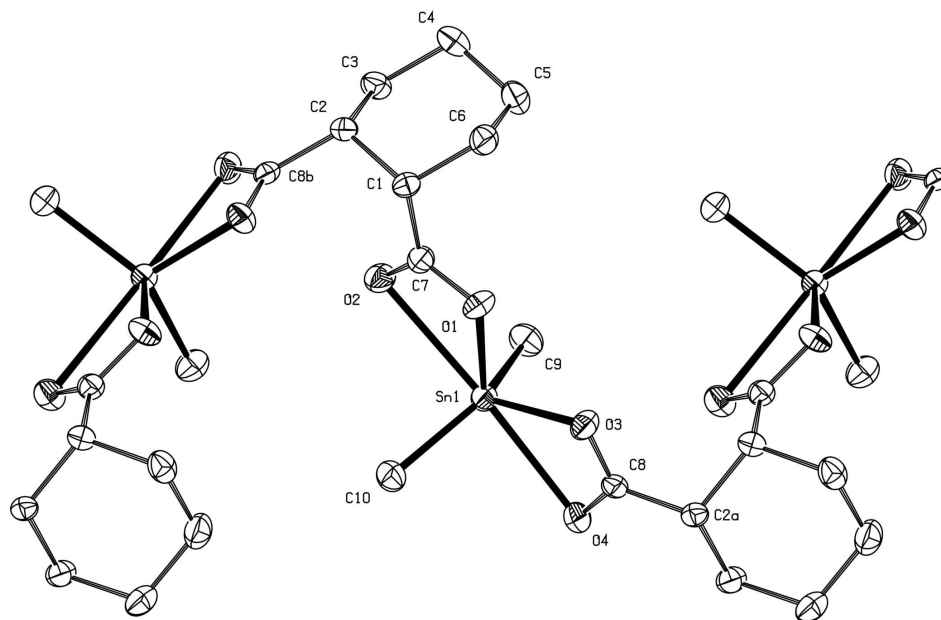
Recently, organotin complexes have been attracting increasing attention partly owing to their determinately or potentially pharmic value, which have been reported many before (Gielen, 2002), and also for the versatile molecular structure and supramolecular architecture exhibited by these complexes (Han *et al.*, 2007). In order to explore the relationships between the properties and structures, we report here the structure of the title complex. Fig. 1 the structure of (I) showing one-dimensional extended polymeric network, and the one-dimensional chain along [001] direction of complex is shown in Fig. 2. Sn atom is six coordinated and displays a octahedral distorted geometry.

**S2. Experimental**

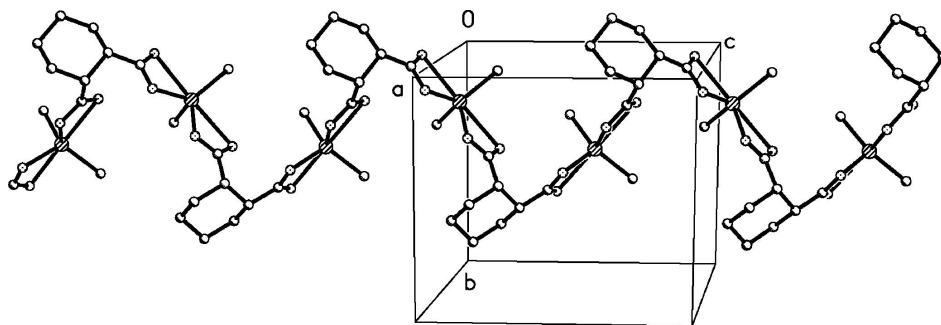
The reaction was carried out under nitrogen atmosphere. *cis*-cyclohexane-1,2-dicarboxylic acid (0.173 g, 1 mmol) was added to the solution of benzene (30 ml) with sodium ethoxide (0.136 g, 2 mmol) in a Schlenk flask. After stirring for 10 min, dimethyltin dichloride (0.220 g, 1 mmol) was added to the mixture. The mixture was kept at 328 K for 12 h. After cooling down to the room temperature, the solution was filtered. The solvent of the filtrate was gradually removed by evaporation under vacuum until a solid product was obtained. The solid was then recrystallized from ether. Colorless single crystals of the title complex were obtained after one week. Yield, 86%. Analysis calculated for C<sub>10</sub>H<sub>16</sub>O<sub>4</sub>Sn: C 48.76, H 6.55; found: C 48.66, H 6.68. The elemental analyses were performed with PERKIN ELMER MODEL 2400 SERIES II.

**S3. Refinement**

All H atoms were placed in geometrically idealized positions methyl (C—H = 0.96 Å), methylene (C—H = 0.97 Å), (C—H = 0.98 Å) and treated as riding on their parent atoms, with  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{CH}_3)$ ,  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{CH}_2)$ ,  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{CH})$ .

**Figure 1**

Part of the structure of (I) showing one-dimensional extended polymeric network, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms have been omitted for clarity. [Symmetry codes: (a)  $x, 1/2 - y, -1/2 + z$ ; (b)  $x, 1/2 - x, 1/2 + z$ ]

**Figure 2**

The one-dimensional zigzag chain of the title complex

### *catena*-Poly[[dimethyltin(IV)]- $\mu$ -*cis*-cyclohexane-1,2-dicarboxylato]

#### Crystal data

[Sn(CH<sub>3</sub>)<sub>2</sub>(C<sub>8</sub>H<sub>10</sub>O<sub>4</sub>)]

$M_r = 318.92$

Monoclinic,  $P2_1/c$

Hall symbol: -P 2ybc

$a = 10.0880(16) \text{ \AA}$

$b = 10.430(2) \text{ \AA}$

$c = 11.592(2) \text{ \AA}$

$\beta = 99.041(2)^\circ$

$V = 1204.5(4) \text{ \AA}^3$

$Z = 4$

$F(000) = 632$

$D_x = 1.759 \text{ Mg m}^{-3}$

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

Cell parameters from 4238 reflections

$\theta = 2.7\text{--}28.3^\circ$

$\mu = 2.11 \text{ mm}^{-1}$

$T = 298 \text{ K}$

Block, colourless

$0.32 \times 0.19 \times 0.17 \text{ mm}$

*Data collection*

Bruker SMART CCD area-detector  
diffractometer  
Radiation source: fine-focus sealed tube  
Graphite monochromator  
 $\varphi$  and  $\omega$  scans  
Absorption correction: multi-scan  
(*SADABS*; Sheldrick, 1996)  
 $T_{\min} = 0.551$ ,  $T_{\max} = 0.715$

6188 measured reflections  
2117 independent reflections  
1822 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.022$   
 $\theta_{\max} = 25.0^\circ$ ,  $\theta_{\min} = 2.0^\circ$   
 $h = -11 \rightarrow 12$   
 $k = -11 \rightarrow 12$   
 $l = -11 \rightarrow 13$

*Refinement*

Refinement on  $F^2$   
Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.031$   
 $wR(F^2) = 0.062$   
 $S = 1.19$   
2117 reflections  
136 parameters  
0 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  
 $w = 1/[\sigma^2(F_o^2) + (0.0181P)^2 + 1.262P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.52 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.43 \text{ e } \text{\AA}^{-3}$

*Special details*

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

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > \sigma(F^2)$  is used only for calculating  $R$ -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

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

|     | <i>x</i>    | <i>y</i>    | <i>z</i>    | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|-------------|-------------|----------------------------------|
| Sn1 | 0.67183 (3) | 0.15366 (3) | 0.10133 (2) | 0.03534 (10)                     |
| O1  | 0.8345 (3)  | 0.2746 (3)  | 0.1623 (2)  | 0.0437 (7)                       |
| O2  | 0.6720 (3)  | 0.3482 (3)  | 0.2497 (3)  | 0.0459 (7)                       |
| O3  | 0.8140 (3)  | 0.0831 (3)  | 0.0045 (2)  | 0.0427 (7)                       |
| O4  | 0.6338 (3)  | -0.0337 (3) | -0.0441 (2) | 0.0448 (7)                       |
| C1  | 0.8867 (4)  | 0.4597 (4)  | 0.2822 (3)  | 0.0333 (9)                       |
| H1  | 0.9542      | 0.4175      | 0.3399      | 0.040*                           |
| C2  | 0.8184 (4)  | 0.5633 (3)  | 0.3471 (3)  | 0.0330 (9)                       |
| H2  | 0.8906      | 0.6163      | 0.3888      | 0.040*                           |
| C3  | 0.7305 (4)  | 0.6523 (4)  | 0.2649 (4)  | 0.0413 (10)                      |
| H3A | 0.6967      | 0.7202      | 0.3095      | 0.050*                           |
| H3B | 0.6542      | 0.6049      | 0.2246      | 0.050*                           |
| C4  | 0.8092 (5)  | 0.7109 (4)  | 0.1751 (4)  | 0.0493 (11)                      |
| H4A | 0.7495      | 0.7637      | 0.1209      | 0.059*                           |
| H4B | 0.8797      | 0.7655      | 0.2149      | 0.059*                           |
| C5  | 0.8706 (5)  | 0.6077 (5)  | 0.1080 (4)  | 0.0548 (12)                      |

|      |            |             |             |             |
|------|------------|-------------|-------------|-------------|
| H5A  | 0.7998     | 0.5575      | 0.0630      | 0.066*      |
| H5B  | 0.9224     | 0.6475      | 0.0539      | 0.066*      |
| C6   | 0.9609 (4) | 0.5204 (4)  | 0.1905 (4)  | 0.0459 (11) |
| H6A  | 1.0366     | 0.5694      | 0.2296      | 0.055*      |
| H6B  | 0.9956     | 0.4531      | 0.1457      | 0.055*      |
| C7   | 0.7883 (4) | 0.3568 (4)  | 0.2295 (3)  | 0.0366 (9)  |
| C8   | 0.7475 (4) | -0.0040 (3) | -0.0606 (3) | 0.0344 (9)  |
| C9   | 0.5448 (4) | 0.2734 (4)  | -0.0107 (4) | 0.0503 (11) |
| H9A  | 0.4773     | 0.3083      | 0.0302      | 0.075*      |
| H9B  | 0.5026     | 0.2249      | -0.0768     | 0.075*      |
| H9C  | 0.5962     | 0.3419      | -0.0369     | 0.075*      |
| C10  | 0.6548 (5) | 0.0271 (4)  | 0.2389 (4)  | 0.0476 (11) |
| H10A | 0.5894     | 0.0594      | 0.2834      | 0.071*      |
| H10B | 0.7401     | 0.0197      | 0.2885      | 0.071*      |
| H10C | 0.6271     | -0.0556     | 0.2077      | 0.071*      |

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

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|-----|--------------|--------------|--------------|--------------|--------------|--------------|
| Sn1 | 0.03871 (17) | 0.03394 (16) | 0.03343 (16) | 0.00183 (14) | 0.00588 (11) | 0.00006 (13) |
| O1  | 0.0425 (16)  | 0.0383 (17)  | 0.0494 (18)  | 0.0012 (14)  | 0.0043 (14)  | -0.0157 (14) |
| O2  | 0.0424 (17)  | 0.0444 (17)  | 0.0523 (18)  | -0.0088 (14) | 0.0116 (14)  | -0.0093 (14) |
| O3  | 0.0429 (16)  | 0.0443 (17)  | 0.0414 (16)  | -0.0032 (14) | 0.0083 (13)  | -0.0124 (13) |
| O4  | 0.0463 (17)  | 0.0475 (17)  | 0.0436 (17)  | -0.0052 (14) | 0.0167 (14)  | 0.0006 (13)  |
| C1  | 0.031 (2)    | 0.030 (2)    | 0.038 (2)    | 0.0017 (17)  | 0.0040 (17)  | -0.0027 (17) |
| C2  | 0.035 (2)    | 0.027 (2)    | 0.036 (2)    | -0.0024 (16) | 0.0042 (17)  | 0.0000 (16)  |
| C3  | 0.042 (2)    | 0.035 (2)    | 0.048 (2)    | 0.003 (2)    | 0.0098 (19)  | 0.0068 (19)  |
| C4  | 0.051 (3)    | 0.044 (3)    | 0.054 (3)    | -0.003 (2)   | 0.011 (2)    | 0.016 (2)    |
| C5  | 0.065 (3)    | 0.058 (3)    | 0.044 (3)    | -0.011 (2)   | 0.018 (2)    | 0.006 (2)    |
| C6  | 0.042 (2)    | 0.049 (3)    | 0.050 (3)    | -0.009 (2)   | 0.018 (2)    | -0.009 (2)   |
| C7  | 0.042 (2)    | 0.034 (2)    | 0.032 (2)    | 0.0055 (19)  | 0.0026 (18)  | 0.0016 (18)  |
| C8  | 0.046 (2)    | 0.025 (2)    | 0.031 (2)    | 0.0025 (18)  | 0.0038 (18)  | 0.0057 (16)  |
| C9  | 0.047 (3)    | 0.051 (3)    | 0.052 (3)    | 0.002 (2)    | 0.003 (2)    | 0.011 (2)    |
| C10 | 0.058 (3)    | 0.045 (3)    | 0.041 (2)    | 0.004 (2)    | 0.009 (2)    | 0.007 (2)    |

*Geometric parameters (Å, °)*

|         |           |                     |           |
|---------|-----------|---------------------|-----------|
| Sn1—O3  | 2.089 (3) | C3—H3A              | 0.9700    |
| Sn1—C9  | 2.089 (4) | C3—H3B              | 0.9700    |
| Sn1—C10 | 2.098 (4) | C4—C5               | 1.516 (6) |
| Sn1—O1  | 2.102 (3) | C4—H4A              | 0.9700    |
| Sn1—O4  | 2.570 (3) | C4—H4B              | 0.9700    |
| Sn1—O2  | 2.660 (3) | C5—C6               | 1.517 (6) |
| O1—C7   | 1.294 (4) | C5—H5A              | 0.9700    |
| O2—C7   | 1.235 (5) | C5—H5B              | 0.9700    |
| O3—C8   | 1.298 (4) | C6—H6A              | 0.9700    |
| O4—C8   | 1.232 (5) | C6—H6B              | 0.9700    |
| C1—C7   | 1.523 (5) | C8—C2 <sup>ii</sup> | 1.509 (5) |

|                        |             |                        |           |
|------------------------|-------------|------------------------|-----------|
| C1—C6                  | 1.530 (5)   | C9—H9A                 | 0.9600    |
| C1—C2                  | 1.540 (5)   | C9—H9B                 | 0.9600    |
| C1—H1                  | 0.9800      | C9—H9C                 | 0.9600    |
| C2—C8 <sup>i</sup>     | 1.509 (5)   | C10—H10A               | 0.9600    |
| C2—C3                  | 1.514 (5)   | C10—H10B               | 0.9600    |
| C2—H2                  | 0.9800      | C10—H10C               | 0.9600    |
| C3—C4                  | 1.532 (6)   |                        |           |
| O3—Sn1—C9              | 106.41 (15) | C5—C4—C3               | 111.2 (4) |
| O3—Sn1—C10             | 109.39 (15) | C5—C4—H4A              | 109.4     |
| C9—Sn1—C10             | 137.14 (18) | C3—C4—H4A              | 109.4     |
| O3—Sn1—O1              | 80.02 (10)  | C5—C4—H4B              | 109.4     |
| C9—Sn1—O1              | 102.72 (15) | C3—C4—H4B              | 109.4     |
| C10—Sn1—O1             | 105.95 (15) | H4A—C4—H4B             | 108.0     |
| O3—Sn1—O4              | 54.83 (10)  | C6—C5—C4               | 110.9 (4) |
| C9—Sn1—O4              | 91.89 (15)  | C6—C5—H5A              | 109.5     |
| C10—Sn1—O4             | 89.93 (14)  | C4—C5—H5A              | 109.5     |
| O1—Sn1—O4              | 134.84 (9)  | C6—C5—H5B              | 109.5     |
| O3—Sn1—O2              | 133.28 (9)  | C4—C5—H5B              | 109.5     |
| C9—Sn1—O2              | 83.34 (15)  | H5A—C5—H5B             | 108.1     |
| C10—Sn1—O2             | 88.85 (14)  | C5—C6—C1               | 112.1 (3) |
| O1—Sn1—O2              | 53.39 (9)   | C5—C6—H6A              | 109.2     |
| O4—Sn1—O2              | 171.54 (9)  | C1—C6—H6A              | 109.2     |
| C7—O1—Sn1              | 105.3 (2)   | C5—C6—H6B              | 109.2     |
| C7—O2—Sn1              | 80.6 (2)    | C1—C6—H6B              | 109.2     |
| C8—O3—Sn1              | 102.9 (2)   | H6A—C6—H6B             | 107.9     |
| C8—O4—Sn1              | 82.2 (2)    | O2—C7—O1               | 120.5 (4) |
| C7—C1—C6               | 111.9 (3)   | O2—C7—C1               | 123.7 (4) |
| C7—C1—C2               | 112.1 (3)   | O1—C7—C1               | 115.8 (3) |
| C6—C1—C2               | 110.8 (3)   | O4—C8—O3               | 119.6 (4) |
| C7—C1—H1               | 107.3       | O4—C8—C2 <sup>ii</sup> | 124.3 (3) |
| C6—C1—H1               | 107.3       | O3—C8—C2 <sup>ii</sup> | 116.1 (3) |
| C2—C1—H1               | 107.3       | Sn1—C9—H9A             | 109.5     |
| C8 <sup>i</sup> —C2—C3 | 113.7 (3)   | Sn1—C9—H9B             | 109.5     |
| C8 <sup>i</sup> —C2—C1 | 110.9 (3)   | H9A—C9—H9B             | 109.5     |
| C3—C2—C1               | 112.7 (3)   | Sn1—C9—H9C             | 109.5     |
| C8 <sup>i</sup> —C2—H2 | 106.3       | H9A—C9—H9C             | 109.5     |
| C3—C2—H2               | 106.3       | H9B—C9—H9C             | 109.5     |
| C1—C2—H2               | 106.3       | Sn1—C10—H10A           | 109.5     |
| C2—C3—C4               | 111.0 (3)   | Sn1—C10—H10B           | 109.5     |
| C2—C3—H3A              | 109.4       | H10A—C10—H10B          | 109.5     |
| C4—C3—H3A              | 109.4       | Sn1—C10—H10C           | 109.5     |
| C2—C3—H3B              | 109.4       | H10A—C10—H10C          | 109.5     |
| C4—C3—H3B              | 109.4       | H10B—C10—H10C          | 109.5     |
| H3A—C3—H3B             | 108.0       |                        |           |

Symmetry codes: (i)  $x, -y+1/2, z+1/2$ ; (ii)  $x, -y+1/2, z-1/2$ .