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Bis(triphenylstannyl) thiophene-2,5-dicarboxylate

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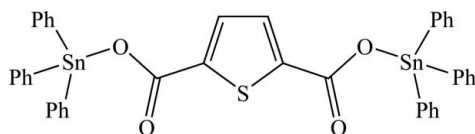
Received 29 April 2009; accepted 27 May 2009

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.016$ Å; disorder in main residue; R factor = 0.052; wR factor = 0.110; data-to-parameter ratio = 13.4.

Molecules of the title compound, $[\text{Sn}_2(\text{C}_6\text{H}_5)_6(\text{C}_6\text{H}_2\text{O}_4\text{S})]$, lie on inversion centres with the central thiophene ring disordered equally over two orientations. The carboxylate groups are approximately coplanar with the thiophene ring [dihedral angle = $4.0(1)^\circ$] and the Sn—O bond distance of $2.058(4)$ Å is comparable to that in related organotin carboxylates.

Related literature

For background literature concerning organotin chemistry, see: Prabusankar & Murugavel (2004); Holmes (1989). For related structures, see: Pellei *et al.* (2008).



Experimental

Crystal data

$[\text{Sn}_2(\text{C}_6\text{H}_5)_6(\text{C}_6\text{H}_2\text{O}_4\text{S})]$
 $M_r = 870.12$
Monoclinic, $P2_1/c$
 $a = 10.1302(10)$ Å
 $b = 18.699(2)$ Å
 $c = 10.3584(11)$ Å
 $\beta = 108.213(2)^\circ$

$V = 1863.8(3)$ Å³
 $Z = 2$
Mo $K\alpha$ radiation
 $\mu = 1.44$ mm⁻¹
 $T = 298$ K
 $0.21 \times 0.11 \times 0.06$ mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.752$, $T_{\max} = 0.919$

9058 measured reflections
3281 independent reflections
2342 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.040$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$
 $wR(F^2) = 0.110$
 $S = 1.02$
3281 reflections

244 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.77$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.63$ e Å⁻³

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 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: BI2371).

References

- Holmes, R. R. (1989). *Acc. Chem. Res.* **22**, 190–197.
Pellei, M., Alidori, S., Benetollo, F., Lobbia, G. G., Mancini, M., Lobbia, G. G. & Santini, C. (2008). *J. Organomet. Chem.* **693**, 996–1004.
Prabusankar, G. & Murugavel, R. (2004). *Organometallics*, **23**, 5644–5647.
Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
Siemens (1996). SMART and SAINT. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

supporting information

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Bis(triphenylstannyl) thiophene-2,5-dicarboxylate

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S1. Comment

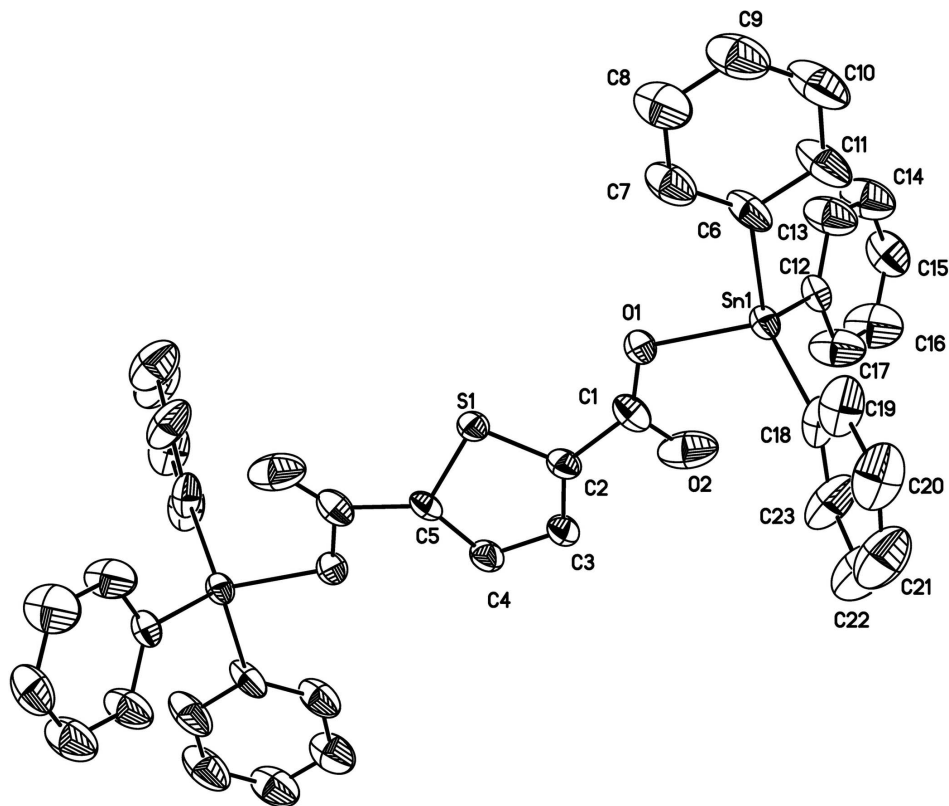
The structural diversity of organotin carboxylates is well recognized and a wide variety of coordination geometries have been reported (Holmes, 1989). It is generally believed that a combination of steric and electronic factors determine the specific structure adapted by a particular organotin carboxylate (Prabusankar & Murugavel, 2004). This is supported through the observation of monomeric, dimeric, tetrameric, oligomeric ladder, cyclic, and drum structures. Furthermore, it has been reported that the size of the carboxylic acids used and the stoichiometry of the reactants play an important role in the formation of solid-state frameworks.

S2. Experimental

The reaction was carried out under a nitrogen atmosphere. Thiophene-2,5-dicarboxylic acid (10 mmol) and sodium ethoxide (20 mmol) were added to a stirred solution of benzene (50 ml) in a three-necked flask and stirred for 0.5 h. Triphenyltin chloride (20 mmol) was then added and the reaction mixture was stirred for 6 h at room temperature. The resulting clear solution was evaporated under vacuum. The product was crystallized from dichloromethane to yield colourless blocks of the title compound. Elemental analysis: calculated C 57.97, H 3.71 %; found: C 57.68, H 3.55 %.

S3. Refinement

H atoms were placed in geometrically idealized positions (C—H = 0.93 Å) and treated as riding on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$.

**Figure 1**

Molecular structure showing 30% probability displacement ellipsoids, with H atoms are omitted. Unlabelled atoms are related to labelled atoms by the symmetry code: $2-x, -y, 1-z$. The symmetry-generated component of the disordered thiophene ring is not shown.

Bis(triphenylstannyl) thiophene-2,5-dicarboxylate

Crystal data

$[\text{Sn}_2(\text{C}_6\text{H}_5)_6(\text{C}_6\text{H}_2\text{O}_4\text{S})]$

$M_r = 870.12$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2ybc$

$a = 10.1302$ (10) Å

$b = 18.699$ (2) Å

$c = 10.3584$ (11) Å

$\beta = 108.213$ (2)°

$V = 1863.8$ (3) Å³

$Z = 2$

$F(000) = 864$

$D_x = 1.550$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 3030 reflections

$\theta = 2.4\text{--}25.2^\circ$

$\mu = 1.44$ mm⁻¹

$T = 298$ K

Needle, colorless

$0.21 \times 0.11 \times 0.06$ mm

Data collection

Bruker SMART APEX CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.752$, $T_{\max} = 0.919$

9058 measured reflections

3281 independent reflections

2342 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.040$

$\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 2.1^\circ$

$h = -12 \rightarrow 6$

$k = -22 \rightarrow 20$

$l = -11 \rightarrow 12$

Refinement

| | |
|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.052$ | H-atom parameters constrained |
| $wR(F^2) = 0.110$ | $w = 1/[\sigma^2(F_o^2) + (0.0214P)^2 + 10.153P]$ |
| $S = 1.02$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 3281 reflections | $(\Delta/\sigma)_{\max} = 0.001$ |
| 244 parameters | $\Delta\rho_{\max} = 0.77 \text{ e } \text{\AA}^{-3}$ |
| 0 restraints | $\Delta\rho_{\min} = -0.63 \text{ e } \text{\AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | |

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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|-----|-------------|-------------|-------------|----------------------------------|-----------|
| Sn1 | 0.66000 (5) | 0.07183 (3) | 0.78299 (5) | 0.04647 (17) | |
| S1 | 0.9299 (4) | 0.0123 (2) | 0.4533 (4) | 0.0479 (9) | 0.50 |
| O1 | 0.7511 (5) | 0.0595 (3) | 0.6325 (5) | 0.0547 (13) | |
| O2 | 0.9220 (7) | 0.0178 (4) | 0.7991 (8) | 0.110 (3) | |
| C1 | 0.8710 (9) | 0.0308 (4) | 0.6808 (10) | 0.061 (2) | |
| C2 | 0.9687 (17) | -0.0005 (9) | 0.6205 (18) | 0.053 (4) | 0.50 |
| C3 | 1.0869 (15) | -0.0332 (8) | 0.6787 (16) | 0.056 (4) | 0.50 |
| H3 | 1.1227 | -0.0425 | 0.7713 | 0.067* | 0.50 |
| C4 | 1.1513 (16) | -0.0519 (8) | 0.5836 (15) | 0.057 (4) | 0.50 |
| H4 | 1.2341 | -0.0775 | 0.6067 | 0.068* | 0.50 |
| C5 | 1.0826 (16) | -0.0293 (8) | 0.4493 (19) | 0.048 (4) | 0.50 |
| C6 | 0.4678 (8) | 0.1119 (5) | 0.6546 (9) | 0.074 (3) | |
| C7 | 0.4344 (10) | 0.1137 (5) | 0.5168 (10) | 0.089 (3) | |
| H7 | 0.4985 | 0.0981 | 0.4752 | 0.107* | |
| C8 | 0.3042 (12) | 0.1388 (6) | 0.4372 (12) | 0.107 (4) | |
| H8 | 0.2812 | 0.1401 | 0.3430 | 0.129* | |
| C9 | 0.2123 (12) | 0.1611 (6) | 0.4998 (14) | 0.113 (4) | |
| H9 | 0.1275 | 0.1798 | 0.4471 | 0.136* | |
| C10 | 0.2392 (11) | 0.1573 (7) | 0.6336 (14) | 0.123 (5) | |
| H10 | 0.1723 | 0.1709 | 0.6731 | 0.147* | |
| C11 | 0.3691 (9) | 0.1327 (6) | 0.7143 (12) | 0.107 (4) | |
| H11 | 0.3894 | 0.1304 | 0.8082 | 0.128* | |
| C12 | 0.7680 (8) | 0.1508 (4) | 0.9236 (7) | 0.0532 (19) | |
| C13 | 0.7151 (11) | 0.2181 (5) | 0.9169 (11) | 0.100 (3) | |
| H13 | 0.6362 | 0.2302 | 0.8457 | 0.120* | |

| | | | | |
|-----|-------------|-------------|-------------|-----------|
| C14 | 0.7770 (12) | 0.2686 (6) | 1.0144 (12) | 0.109 (4) |
| H14 | 0.7375 | 0.3138 | 1.0089 | 0.131* |
| C15 | 0.8898 (11) | 0.2541 (6) | 1.1140 (11) | 0.091 (3) |
| H15 | 0.9309 | 0.2884 | 1.1793 | 0.109* |
| C16 | 0.9439 (12) | 0.1896 (7) | 1.1196 (12) | 0.121 (4) |
| H16 | 1.0244 | 0.1788 | 1.1899 | 0.145* |
| C17 | 0.8848 (11) | 0.1373 (5) | 1.0241 (10) | 0.102 (4) |
| H17 | 0.9267 | 0.0927 | 1.0301 | 0.123* |
| C18 | 0.6293 (11) | -0.0309 (5) | 0.8534 (9) | 0.072 (3) |
| C19 | 0.4946 (13) | -0.0581 (6) | 0.8128 (10) | 0.101 (3) |
| H19 | 0.4219 | -0.0301 | 0.7600 | 0.121* |
| C20 | 0.4668 (15) | -0.1284 (7) | 0.8514 (12) | 0.115 (4) |
| H20 | 0.3770 | -0.1465 | 0.8293 | 0.138* |
| C21 | 0.5790 (17) | -0.1672 (7) | 0.9219 (13) | 0.127 (5) |
| H21 | 0.5634 | -0.2144 | 0.9418 | 0.152* |
| C22 | 0.7120 (16) | -0.1434 (7) | 0.9663 (12) | 0.130 (5) |
| H22 | 0.7840 | -0.1725 | 1.0172 | 0.156* |
| C23 | 0.7362 (14) | -0.0732 (6) | 0.9321 (10) | 0.110 (4) |
| H23 | 0.8258 | -0.0547 | 0.9629 | 0.132* |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|------------|------------|------------|-------------|-------------|-------------|
| Sn1 | 0.0405 (3) | 0.0537 (3) | 0.0483 (3) | 0.0019 (3) | 0.0182 (2) | -0.0062 (3) |
| S1 | 0.040 (2) | 0.052 (2) | 0.054 (3) | 0.0089 (19) | 0.0192 (19) | 0.0058 (19) |
| O1 | 0.050 (3) | 0.062 (3) | 0.060 (3) | 0.008 (3) | 0.028 (3) | 0.005 (3) |
| O2 | 0.092 (5) | 0.082 (5) | 0.118 (6) | 0.012 (4) | -0.023 (5) | 0.006 (4) |
| C1 | 0.048 (5) | 0.061 (5) | 0.080 (6) | -0.002 (4) | 0.026 (5) | -0.017 (5) |
| C2 | 0.042 (10) | 0.055 (10) | 0.062 (11) | 0.012 (8) | 0.018 (9) | 0.000 (8) |
| C3 | 0.049 (9) | 0.065 (10) | 0.057 (10) | 0.017 (8) | 0.020 (8) | 0.002 (8) |
| C4 | 0.046 (9) | 0.064 (10) | 0.059 (10) | 0.015 (8) | 0.015 (8) | -0.002 (8) |
| C5 | 0.040 (9) | 0.047 (10) | 0.064 (12) | 0.010 (8) | 0.025 (9) | -0.005 (9) |
| C6 | 0.053 (5) | 0.082 (6) | 0.079 (6) | 0.014 (5) | 0.010 (5) | -0.042 (5) |
| C7 | 0.069 (6) | 0.092 (7) | 0.090 (7) | 0.017 (5) | 0.003 (6) | -0.041 (6) |
| C8 | 0.087 (8) | 0.110 (9) | 0.099 (8) | 0.020 (7) | -0.008 (7) | -0.038 (7) |
| C9 | 0.080 (8) | 0.111 (9) | 0.121 (10) | 0.026 (7) | -0.010 (8) | -0.038 (8) |
| C10 | 0.074 (7) | 0.142 (11) | 0.134 (11) | 0.040 (7) | 0.006 (8) | -0.056 (9) |
| C11 | 0.060 (6) | 0.134 (9) | 0.111 (8) | 0.032 (6) | 0.007 (6) | -0.055 (7) |
| C12 | 0.050 (4) | 0.061 (5) | 0.055 (5) | -0.002 (4) | 0.025 (4) | -0.011 (4) |
| C13 | 0.090 (7) | 0.081 (7) | 0.104 (8) | 0.019 (6) | -0.004 (6) | -0.039 (6) |
| C14 | 0.098 (8) | 0.086 (7) | 0.119 (9) | 0.012 (7) | -0.001 (8) | -0.043 (7) |
| C15 | 0.082 (7) | 0.092 (8) | 0.092 (8) | -0.016 (6) | 0.019 (6) | -0.046 (6) |
| C16 | 0.099 (9) | 0.110 (9) | 0.111 (9) | 0.002 (8) | -0.031 (7) | -0.031 (8) |
| C17 | 0.089 (7) | 0.081 (7) | 0.096 (8) | 0.015 (6) | -0.030 (6) | -0.024 (6) |
| C18 | 0.090 (7) | 0.079 (6) | 0.053 (5) | -0.033 (6) | 0.033 (5) | -0.012 (5) |
| C19 | 0.121 (9) | 0.104 (8) | 0.080 (7) | -0.043 (7) | 0.035 (7) | -0.015 (6) |
| C20 | 0.130 (11) | 0.116 (10) | 0.096 (9) | -0.064 (9) | 0.033 (8) | -0.014 (7) |
| C21 | 0.149 (13) | 0.119 (11) | 0.102 (10) | -0.045 (10) | 0.023 (10) | 0.019 (8) |

| | | | | | | |
|-----|------------|------------|-----------|------------|-----------|-----------|
| C22 | 0.151 (13) | 0.113 (10) | 0.103 (9) | -0.036 (9) | 0.006 (9) | 0.020 (8) |
| C23 | 0.146 (11) | 0.092 (8) | 0.072 (7) | -0.044 (8) | 0.006 (7) | 0.019 (6) |

Geometric parameters (Å, °)

| | | | |
|--------------------|------------|-------------|------------|
| Sn1—O1 | 2.058 (4) | C10—H10 | 0.930 |
| Sn1—C18 | 2.112 (9) | C11—H11 | 0.930 |
| Sn1—C6 | 2.121 (9) | C12—C17 | 1.333 (11) |
| Sn1—C12 | 2.122 (7) | C12—C13 | 1.359 (11) |
| S1—C2 | 1.669 (18) | C13—C14 | 1.382 (12) |
| S1—C5 | 1.744 (15) | C13—H13 | 0.930 |
| O1—C1 | 1.279 (9) | C14—C15 | 1.307 (13) |
| O2—C1 | 1.197 (10) | C14—H14 | 0.930 |
| C1—C2 | 1.449 (17) | C15—C16 | 1.320 (14) |
| C2—C3 | 1.31 (2) | C15—H15 | 0.930 |
| C3—C4 | 1.385 (19) | C16—C17 | 1.386 (13) |
| C3—H3 | 0.930 | C16—H16 | 0.930 |
| C4—C5 | 1.41 (2) | C17—H17 | 0.930 |
| C4—H4 | 0.930 | C18—C23 | 1.382 (14) |
| C5—C1 ⁱ | 1.560 (19) | C18—C19 | 1.392 (13) |
| C6—C7 | 1.361 (12) | C19—C20 | 1.428 (14) |
| C6—C11 | 1.386 (12) | C19—H19 | 0.930 |
| C7—C8 | 1.400 (13) | C20—C21 | 1.354 (16) |
| C7—H7 | 0.930 | C20—H20 | 0.930 |
| C8—C9 | 1.356 (15) | C21—C22 | 1.355 (16) |
| C8—H8 | 0.930 | C21—H21 | 0.930 |
| C9—C10 | 1.328 (15) | C22—C23 | 1.402 (14) |
| C9—H9 | 0.930 | C22—H22 | 0.930 |
| C10—C11 | 1.399 (13) | C23—H23 | 0.930 |
| O1—Sn1—C18 | 108.0 (3) | C6—C11—H11 | 120.0 |
| O1—Sn1—C6 | 96.1 (3) | C10—C11—H11 | 120.0 |
| C18—Sn1—C6 | 109.4 (4) | C17—C12—C13 | 117.1 (8) |
| O1—Sn1—C12 | 109.9 (2) | C17—C12—Sn1 | 123.0 (6) |
| C18—Sn1—C12 | 119.7 (3) | C13—C12—Sn1 | 119.9 (6) |
| C6—Sn1—C12 | 111.1 (3) | C12—C13—C14 | 120.9 (9) |
| C2—S1—C5 | 92.1 (7) | C12—C13—H13 | 119.6 |
| C1—O1—Sn1 | 110.3 (5) | C14—C13—H13 | 119.6 |
| O2—C1—O1 | 122.7 (8) | C15—C14—C13 | 121.4 (10) |
| O2—C1—C2 | 103.0 (11) | C15—C14—H14 | 119.3 |
| O1—C1—C2 | 134.0 (11) | C13—C14—H14 | 119.3 |
| C3—C2—C1 | 129.7 (16) | C14—C15—C16 | 118.2 (10) |
| C3—C2—S1 | 115.5 (12) | C14—C15—H15 | 120.9 |
| C1—C2—S1 | 114.7 (12) | C16—C15—H15 | 120.9 |
| C2—C3—C4 | 110.8 (15) | C15—C16—C17 | 122.2 (10) |
| C2—C3—H3 | 124.6 | C15—C16—H16 | 118.9 |
| C4—C3—H3 | 124.6 | C17—C16—H16 | 118.9 |
| C3—C4—C5 | 115.4 (15) | C12—C17—C16 | 120.2 (10) |

| | | | |
|------------------------|------------|-------------|------------|
| C3—C4—H4 | 122.3 | C12—C17—H17 | 119.9 |
| C5—C4—H4 | 122.3 | C16—C17—H17 | 119.9 |
| C4—C5—S1 | 106.1 (13) | C23—C18—C19 | 118.9 (9) |
| C1 ⁱ —C5—S1 | 122.5 (12) | C23—C18—Sn1 | 123.4 (7) |
| C7—C6—C11 | 118.9 (9) | C19—C18—Sn1 | 117.6 (8) |
| C7—C6—Sn1 | 123.0 (6) | C18—C19—C20 | 120.8 (12) |
| C11—C6—Sn1 | 117.9 (7) | C18—C19—H19 | 119.6 |
| C6—C7—C8 | 120.4 (10) | C20—C19—H19 | 119.6 |
| C6—C7—H7 | 119.8 | C21—C20—C19 | 116.0 (12) |
| C8—C7—H7 | 119.8 | C21—C20—H20 | 122.0 |
| C9—C8—C7 | 118.9 (11) | C19—C20—H20 | 122.0 |
| C9—C8—H8 | 120.6 | C20—C21—C22 | 125.8 (13) |
| C7—C8—H8 | 120.6 | C20—C21—H21 | 117.1 |
| C10—C9—C8 | 122.3 (11) | C22—C21—H21 | 117.1 |
| C10—C9—H9 | 118.8 | C21—C22—C23 | 117.1 (13) |
| C8—C9—H9 | 118.8 | C21—C22—H22 | 121.5 |
| C9—C10—C11 | 119.3 (12) | C23—C22—H22 | 121.5 |
| C9—C10—H10 | 120.4 | C18—C23—C22 | 121.2 (12) |
| C11—C10—H10 | 120.4 | C18—C23—H23 | 119.4 |
| C6—C11—C10 | 120.1 (11) | C22—C23—H23 | 119.4 |

Symmetry code: (i) $-x+2, -y, -z+1$.