

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

# Bis(2-ethoxycarbonyl-ethyl- $\kappa^2C^1,O$ )(2-thioxo-1,3-dithiole-4,5-dithiolato- $\kappa^2S^4,S^5$ )tin(IV)

 Geraldo M. de Lima,<sup>a</sup> Solange M. S. V. Wardell,<sup>b</sup> James L. Wardell<sup>a‡</sup> and Edward R. T. Tiekink<sup>c\*</sup>

<sup>a</sup>Departamento de Química, ICEx, Universidade Federal de Minas Gerais, 31270-901 Belo Horizonte, MG, Brazil, <sup>b</sup>CHEMSOL, 1 Harcourt Road, Aberdeen AB15 5NY, Scotland, and <sup>c</sup>Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: edward.tiekink@gmail.com

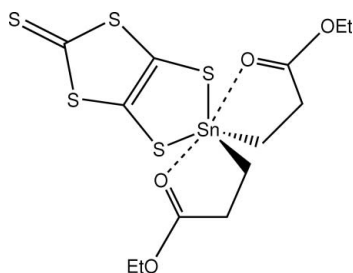
Received 11 November 2009; accepted 17 November 2009

 Key indicators: single-crystal X-ray study;  $T = 120$  K; mean  $\sigma(C-C) = 0.007$  Å;  $R$  factor = 0.029;  $wR$  factor = 0.064; data-to-parameter ratio = 18.5.

In the title compound,  $[Sn(C_5H_9O_2)_2(C_3S_5)]$ , the immediate environment around the Sn centre is defined by two S and two C atoms that define an approximately tetrahedral geometry. The close approach of the pendant carbonyl O atoms [ $Sn-O = 2.577(3)$  and  $2.573(3)$  Å] increases the coordination number to six. Supramolecular chains are formed along the  $a$  axis in the crystal structure owing to the presence of  $C-H \cdots O$  contacts.

## Related literature

For original industrial interest in functionally substituted-alkyl-tin compounds, see: Lanigen & Weinberg (1976). For studies concerning the coordination chemistry of functionally substituted-alkyl-tin compounds, see: Harrison *et al.* (1979); Balasubramanian *et al.* (1997); Milne *et al.* (2005); Tian *et al.* (2005); de Lima *et al.* (2009). For related structures of functionally substituted-alkyl-tin compounds, see: Buchanan *et al.* (1996); Howie & Wardell, (2001). For the synthesis, see: Hutton & Oakes (1976); Valade *et al.* (1985).



‡ Additional correspondence author, e-mail: j.wardell@abdn.ac.uk.

## Experimental

## Crystal data

$[Sn(C_5H_9O_2)_2(C_3S_5)]$   
 $M_r = 517.26$   
 Orthorhombic,  $Pna2_1$   
 $a = 12.1224(2)$  Å  
 $b = 13.3825(2)$  Å  
 $c = 11.9228(2)$  Å

$V = 1934.21(5)$  Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 1.87$  mm<sup>-1</sup>  
 $T = 120$  K  
 $0.25 \times 0.10 \times 0.08$  mm

## Data collection

Bruker-Nonius 95mm CCD camera  
 on  $\kappa$ -goniostat diffractometer  
 Absorption correction: multi-scan  
 (SADABS; Sheldrick, 2003)  
 $T_{min} = 0.025$ ,  $T_{max} = 0.052$

14014 measured reflections  
 3895 independent reflections  
 3457 reflections with  $I > 2\sigma(I)$   
 $R_{int} = 0.056$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$   
 $wR(F^2) = 0.064$   
 $S = 1.04$   
 3895 reflections  
 211 parameters

15 restraints  
 H-atom parameters constrained  
 $\Delta\rho_{max} = 0.64$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -0.48$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C12-H12a \cdots O1^i$	0.99	2.38	3.338 (6)	164
$C7-H7a \cdots O3^{ii}$	0.99	2.46	3.450 (7)	178

 Symmetry codes: (i)  $x - \frac{1}{2}, -y + \frac{1}{2}, z$ ; (ii)  $x + \frac{1}{2}, -y + \frac{1}{2}, z$ .

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2009).

The use of the EPSRC X-ray crystallographic service at the University of Southampton, England, and the valuable assistance of the staff there is gratefully acknowledged. JLW acknowledges support from FAPEMIG (Brazil).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2162).

## References

- Balasubramanian, R., Chohan, Z. H., Doidge-Harrison, S. M. S. V., Howie, R. A. & Wardell, J. L. (1997). *Polyhedron*, **16**, 4283–4295.  
 Brandenburg, K. (2006). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.  
 Buchanan, H., Howie, R. A., Khan, A., Spencer, G. M., Wardell, J. L. & Atupers, J. H. (1996). *J. Chem. Soc. Dalton Trans.* pp. 541–548.  
 Harrison, P. G., King, T. J. & Healey, M. A. (1979). *J. Organomet. Chem.* **182**, 17–36.  
 Hooft, R. W. W. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.  
 Howie, R. A. & Wardell, J. L. (2001). *Acta Cryst.* **C57**, 1041–1043.  
 Hutton, R. E. & Oakes, V. (1976). *Adv. Chem. Ser.* **157**, 123–133.  
 Lanigen, D. & Weinberg, E. L. (1976). *Adv. Chem. Ser.* **157**, 134–142.  
 Lima, G. M. de, Milne, B. F., Pereira, R. P., Rocco, A. M., Skakle, J. M., Travis, A. J., Wardell, J. L. & Wardell, S. M. S. V. (2009). *J. Mol. Struct.* **921**, 244–250.  
 Milne, B. F., Pereira, R. P., Rocco, A. M., Skakle, J. M. S., Travis, A. J., Wardell, J. L. & Wardell, S. M. S. V. (2005). *Appl. Organomet. Chem.* **19**, 363–371.

- Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
- Sheldrick, G. M. (2003). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Tian, L. J., Zhang, L. P., Liu, X. C. & Zhou, Z. Y. (2005). *Appl. Organomet. Chem.* **19**, 198–199.
- Valade, L., Legros, J. P., Bousseau, M., Garbaukas, M. & Interrante, L. V. (1985). *J. Chem. Soc. Dalton Trans.* pp. 783–794.
- Westrip, S. P. (2009). *publCIF*. In preparation.

**supplementary materials**

*Acta Cryst.* (2009). E65, m1635-m1636 [ doi:10.1107/S1600536809048971 ]

## Bis(2-ethoxycarbonylethyl- $\kappa^2C^1,O$ )(2-thioxo-1,3-dithiole-4,5-dithiolato- $\kappa^2S^4,S^5$ )tin(IV)

G. M. de Lima, S. M. S. V. Wardell, J. L. Wardell and E. R. T. Tiekink

### Comment

Functionally substituted-alkyl-tin compounds,  $X_3SnCR_2CH_2CO_2R'$  and  $X_2Sn(CR_2CH_2CO_2R')_2$  ( $X$  = halide,  $R$  = H or alkyl;  $R'$  = alkyl or aryl), are readily available from reactions first reported in the 1970's (Hutton & Oakes, 1976), starting from  $R_2CCHCOY$  ( $Y = R'$  or  $OR'$ ),  $HX$  and  $SnX_2$  (for  $X_3SnCR_2CH_2COY$  compounds) or  $HX$  and tin (for  $X_2Sn(CR_2CH_2COY)_2$  substrates). Original interest with these compounds was primarily concerned with their industrial potential as precursors of PVC stabilizers (Lanigen & Weinberg, 1976) but also with regard to their coordination chemistry. Although the potential for use in PVC stabilization has not been realised commercially, the interest in the coordination chemistry, generally of compounds containing  $SnCR_2CH_2COY$  moieties, has been maintained over the succeeding decades. Particular interest has been paid to coordination modes of the  $CR_2CH_2COY$  ligands (de Lima *et al.* 2009; Tian *et al.*, 2005; Milne *et al.*, 2005; Harrison *et al.*, 1979). Diester-tin compounds,  $(MeO_2CCH_2CH_2)_2SnX_2$  ( $X$  = halide or thiocyanate) (Balasubramanian *et al.*, 1997; Harrison *et al.*, 1979) and  $(MeO_2CCH_2CH_2)_2Sn(dmit)$  ( $dmit = 1,3$ -dithiole-2-thione-4,5-dithiolato; Buchanan *et al.*, 1996) have been shown to be molecular species with hexa-coordinate tin centres both in the solid-state and in non-coordinating solutions, as a consequence of the ( $C,O$ )-chelating ligands. Compounds  $(MeCOCH_2CMe_2)SnX_2$  also contain hexa-co-ordinate tin centres ( $X = Cl$  or  $dmit$ ; Howie & Wardell, 2001).

The molecular structure of (I) features a chelating  $dmit$  ligand as well as two C-bound  $CH_2CH_2CO_2Et$  ligands, each of which coordinates *via* the  $\alpha$  carbon atom. The Sn atom exists within a distorted tetrahedral  $C_2S_2$  donor set, Fig. 1. Significant distortions from the ideal geometry arise from the close approach of two carbonyl-O atoms [ $Sn-O = 2.577$  (3) and 2.573 (3) Å] thereby increasing the coordination number to six. The expanded geometry is therefore based on a highly distorted octahedron. The  $dmit$  ligand forms nearly equivalent Sn–S bond distances of 2.4805 (11) and 2.4958 (9) Å. In many respects, the molecular structure of (I) resembles that of the previously reported methyl ester analogue (Buchanan *et al.* (1996). The former has crystallographic twofold symmetry which is absent in (I) owing to a misalignment of the ethyl substituents.

In the crystal structure, molecules are connected into a supramolecular chain along the  $a$  axis *via* C–H $\cdots$ O interactions, with each molecule forming two donor and two acceptor contacts, Table 1 and Fig. 2.

### Experimental

Solutions of  $Cl_2Sn(CH_2CH_2CO_2Et)_2$  (0.75 g, 2 mmol) (Hutton & Oakes, 1976) in acetone (20 ml) and  $[NEt_4]_2[Zn(dmit)_2]$  (0.70 g, 1 mmol) (Valade *et al.*, 1985) in acetone (20 ml) were mixed and the reaction mixture was maintained at room temperature. After 1 h, the reaction mixture was filtered and the filtrate evaporated to leave a solid residue, which after washing with water, was crystallized from acetone to give the title compound as a red-coloured crystalline solid, m.pt. 394–396 K.  $^1H$  NMR ( $CDCl_3$ )  $\delta$ : 1.25 [t, 3H,  $J(1H-1H) = 7.2$  Hz, Me], 1.93 (t, 2H,  $J(1H-1H) = 7.2$  Hz,  $J(^{119}Sn-1H) = 84.2$  Hz),  $CH_2Sn$ ), 2.96 (t, 2H,  $J(1H-1H) = 7.2$  Hz,  $J(^{119}Sn-1H) = 137.6$  Hz,  $CH_2CH_2Sn$ ), 4.20 (q, 2H,  $J(1H-1H) = 7.2$  Hz,  $OCH_2$ ) p.p.m.  $^{13}C$  NMR ( $CDCl_3$ , 62.9 MHz)  $\delta$ : 13.9 ( $CH_3$ ), 22.8 [ $J(^{119}Sn-^{13}C) = 580$  Hz,  $CH_2Sn$ ], 28.5 [ $J(^{119}Sn-^{13}C) = 46$  Hz,

## supplementary materials

CH<sub>2</sub>CH<sub>2</sub>Sn), 63.6 (OCH<sub>2</sub>), 129.8 (CC), 181.3 (CO), 210.6 (C S) p.p.m. <sup>119</sup>Sn (CD<sub>2</sub>Cl<sub>2</sub>, 93.3 MHz) δ: 84.2 p.p.m. IR (KBr): 1680 (νCO), 1437 (νCC), 1031 (νCS), 890 (νC-S), 465 (νC-S) cm<sup>-1</sup>.

### Refinement

All H atoms were geometrically placed (C–H = 0.98–0.99 Å) and refined as riding with  $U_{iso}(H) = 1.2-1.5U_{eq}(C)$ . Indications for disorder was found in the O2-ethyl group. Multiple sites could not be resolved, however. The O2—C7 and C7—C8 bond distances were refined with the distance restraints of  $1.460 \pm 0.005$  Å and  $1.500 \pm 0.005$  Å, respectively. Further, their anisotropic displacement parameters were constrained to be isotropic with the ISOR command in *SHELXL97* (Sheldrick, 2008). The structure was refined as a racemic twin precluding the determination of the absolute structure.

### Figures

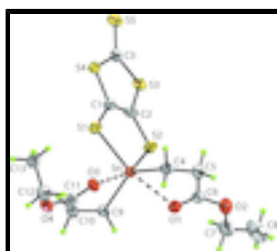


Fig. 1. Molecular structure of (I) showing atom-labelling scheme and displacement ellipsoids at the 50% probability level.

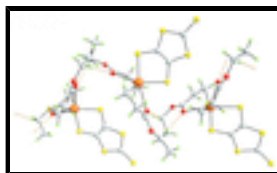


Fig. 2. Supramolecular chain formation along the *a* axis in (I) mediated by C–H...O contacts (orange dashed lines).

### Bis(2-ethoxycarbonyl-ethyl-κ<sup>2</sup>C<sup>1</sup>,O)(2-thioxo-1,3-dithiole-4,5-dithiolato-κ<sup>2</sup>S<sup>4</sup>,S<sup>5</sup>)tin(IV)

#### Crystal data

[Sn(C<sub>5</sub>H<sub>9</sub>O<sub>2</sub>)<sub>2</sub>(C<sub>3</sub>S<sub>5</sub>)]

$M_r = 517.26$

Orthorhombic, *Pna*2<sub>1</sub>

Hall symbol: P 2c -2n

$a = 12.1224$  (2) Å

$b = 13.3825$  (2) Å

$c = 11.9228$  (2) Å

$V = 1934.21$  (5) Å<sup>3</sup>

$Z = 4$

$F_{000} = 1032$

$D_x = 1.776$  Mg m<sup>-3</sup>

Mo *K*α radiation,  $\lambda = 0.71069$  Å

Cell parameters from 16139 reflections

$\theta = 2.9-27.5^\circ$

$\mu = 1.87$  mm<sup>-1</sup>

$T = 120$  K

Block, orange

$0.25 \times 0.10 \times 0.08$  mm

#### Data collection

Bruker–Nonius 95mm CCD camera on κ-goniostat diffractometer

3895 independent reflections

Radiation source: Bruker-Nonius FR591 rotating anode	3457 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.056$
Detector resolution: 9.091 pixels $\text{mm}^{-1}$	$\theta_{\text{max}} = 27.5^\circ$
$T = 120$ K	$\theta_{\text{min}} = 3.0^\circ$
$\varphi$ and $\omega$ scans	$h = -15 \rightarrow 14$
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	$k = -17 \rightarrow 17$
$T_{\text{min}} = 0.025$ , $T_{\text{max}} = 0.052$	$l = -13 \rightarrow 15$
14014 measured reflections	

### Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.029$	H-atom parameters constrained
$wR(F^2) = 0.064$	$w = 1/[\sigma^2(F_o^2) + (0.0188P)^2 + 0.7375P]$
$S = 1.04$	where $P = (F_o^2 + 2F_c^2)/3$
3895 reflections	$(\Delta/\sigma)_{\text{max}} = 0.002$
211 parameters	$\Delta\rho_{\text{max}} = 0.64 \text{ e } \text{\AA}^{-3}$
15 restraints	$\Delta\rho_{\text{min}} = -0.48 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

### Special details

**Geometry.** All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
Sn	0.529829 (17)	0.337064 (17)	0.45100 (3)	0.02754 (8)
S1	0.53453 (8)	0.51943 (8)	0.41409 (10)	0.0335 (3)
S2	0.73501 (8)	0.33979 (7)	0.46792 (12)	0.0345 (3)
S3	0.88592 (8)	0.51390 (8)	0.48204 (9)	0.0346 (3)
S4	0.72095 (7)	0.66289 (7)	0.43844 (12)	0.0321 (2)
S5	0.94961 (10)	0.72872 (9)	0.49178 (10)	0.0453 (3)
O1	0.5826 (3)	0.1584 (2)	0.5112 (3)	0.0379 (7)

## supplementary materials

---

O2	0.6277 (3)	0.0814 (3)	0.6703 (3)	0.0573 (10)
O3	0.3343 (2)	0.3912 (2)	0.3931 (2)	0.0309 (6)
O4	0.2446 (2)	0.4120 (2)	0.2305 (2)	0.0360 (7)
C1	0.6756 (3)	0.5390 (3)	0.4401 (4)	0.0295 (9)
C2	0.7527 (3)	0.4689 (3)	0.4608 (5)	0.0307 (8)
C3	0.8572 (3)	0.6406 (3)	0.4716 (4)	0.0313 (10)
C4	0.4687 (4)	0.3126 (4)	0.6175 (4)	0.0356 (11)
H4A	0.4635	0.3773	0.6572	0.043*
H4B	0.3938	0.2834	0.6134	0.043*
C5	0.5442 (4)	0.2423 (4)	0.6830 (4)	0.0417 (11)
H5A	0.6080	0.2807	0.7119	0.050*
H5B	0.5033	0.2154	0.7483	0.050*
C6	0.5855 (4)	0.1571 (3)	0.6132 (4)	0.0412 (11)
C7	0.6762 (6)	0.0013 (4)	0.6013 (5)	0.0691 (19)
H7A	0.7202	0.0313	0.5399	0.083*
H7B	0.6164	-0.0389	0.5671	0.083*
C8	0.7474 (5)	-0.0643 (6)	0.6694 (5)	0.086 (2)
H8A	0.8081	-0.0250	0.7012	0.129*
H8B	0.7776	-0.1175	0.6220	0.129*
H8C	0.7040	-0.0939	0.7302	0.129*
C9	0.4905 (3)	0.2620 (3)	0.2973 (3)	0.0314 (10)
H9A	0.5593	0.2486	0.2552	0.038*
H9B	0.4553	0.1970	0.3145	0.038*
C10	0.4131 (4)	0.3238 (3)	0.2247 (4)	0.0367 (10)
H10A	0.4569	0.3730	0.1817	0.044*
H10B	0.3758	0.2792	0.1704	0.044*
C11	0.3272 (3)	0.3782 (3)	0.2923 (4)	0.0301 (9)
C12	0.1619 (4)	0.4726 (4)	0.2870 (4)	0.0392 (11)
H12A	0.1468	0.4441	0.3621	0.047*
H12B	0.0924	0.4710	0.2434	0.047*
C13	0.1992 (4)	0.5781 (3)	0.2994 (4)	0.0445 (11)
H13A	0.2706	0.5796	0.3379	0.067*
H13B	0.1448	0.6156	0.3433	0.067*
H13C	0.2068	0.6086	0.2250	0.067*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Sn	0.02877 (12)	0.02691 (13)	0.02694 (13)	-0.00144 (10)	0.00049 (14)	-0.00137 (15)
S1	0.0284 (5)	0.0272 (5)	0.0451 (7)	0.0013 (4)	-0.0039 (4)	0.0017 (4)
S2	0.0292 (4)	0.0272 (5)	0.0472 (9)	0.0016 (4)	-0.0027 (5)	0.0020 (6)
S3	0.0289 (4)	0.0381 (5)	0.0368 (7)	-0.0026 (4)	-0.0014 (4)	-0.0022 (5)
S4	0.0363 (4)	0.0271 (4)	0.0330 (7)	-0.0037 (4)	0.0001 (5)	-0.0019 (5)
S5	0.0491 (6)	0.0502 (7)	0.0367 (6)	-0.0208 (6)	0.0015 (5)	-0.0058 (6)
O1	0.0438 (18)	0.0333 (16)	0.0365 (19)	0.0018 (13)	-0.0047 (14)	-0.0023 (13)
O2	0.075 (3)	0.049 (2)	0.048 (2)	0.0012 (18)	-0.0188 (18)	0.0071 (18)
O3	0.0315 (13)	0.0359 (16)	0.0254 (16)	0.0003 (13)	-0.0035 (12)	-0.0002 (13)
O4	0.0329 (15)	0.0445 (19)	0.0306 (18)	0.0062 (14)	-0.0022 (13)	-0.0018 (15)

C1	0.0323 (17)	0.0266 (17)	0.030 (2)	-0.0050 (15)	-0.003 (2)	0.001 (2)
C2	0.0297 (16)	0.0280 (18)	0.034 (2)	-0.0046 (14)	-0.002 (2)	-0.009 (2)
C3	0.0352 (18)	0.038 (2)	0.021 (3)	-0.0066 (16)	0.0031 (17)	0.0026 (19)
C4	0.039 (3)	0.035 (3)	0.032 (3)	-0.0048 (19)	0.0015 (18)	-0.004 (2)
C5	0.051 (3)	0.047 (3)	0.027 (2)	-0.010 (2)	-0.001 (2)	0.004 (2)
C6	0.042 (3)	0.039 (3)	0.043 (3)	-0.014 (2)	-0.005 (2)	0.008 (2)
C7	0.092 (5)	0.051 (4)	0.065 (4)	0.018 (3)	-0.031 (4)	-0.016 (3)
C8	0.069 (4)	0.121 (7)	0.069 (5)	0.021 (4)	-0.012 (3)	-0.022 (4)
C9	0.034 (2)	0.031 (2)	0.029 (2)	0.0001 (19)	0.0013 (18)	-0.002 (2)
C10	0.036 (2)	0.046 (3)	0.028 (2)	0.006 (2)	-0.003 (2)	-0.003 (2)
C11	0.031 (2)	0.027 (2)	0.032 (3)	0.0007 (18)	-0.0012 (18)	0.0004 (18)
C12	0.032 (2)	0.048 (3)	0.038 (3)	0.007 (2)	0.0047 (19)	-0.002 (2)
C13	0.044 (3)	0.038 (3)	0.051 (3)	0.005 (2)	0.001 (2)	0.006 (2)

*Geometric parameters (Å, °)*

Sn—C4	2.144 (5)	C4—H4B	0.9900
Sn—C9	2.144 (4)	C5—C6	1.498 (7)
Sn—S1	2.4805 (11)	C5—H5A	0.9900
Sn—S2	2.4958 (9)	C5—H5B	0.9900
Sn—O3	2.573 (3)	C7—C8	1.475 (4)
Sn—O1	2.577 (3)	C7—H7A	0.9900
S1—C1	1.758 (4)	C7—H7B	0.9900
S2—C2	1.743 (4)	C8—H8A	0.9800
S3—C3	1.735 (4)	C8—H8B	0.9800
S3—C2	1.742 (3)	C8—H8C	0.9800
S4—C3	1.724 (4)	C9—C10	1.520 (6)
S4—C1	1.746 (4)	C9—H9A	0.9900
S5—C3	1.644 (4)	C9—H9B	0.9900
O1—C6	1.217 (5)	C10—C11	1.505 (6)
O2—C6	1.324 (6)	C10—H10A	0.9900
O2—C7	1.474 (4)	C10—H10B	0.9900
O3—C11	1.217 (5)	C12—C13	1.490 (6)
O4—C11	1.323 (5)	C12—H12A	0.9900
O4—C12	1.454 (5)	C12—H12B	0.9900
C1—C2	1.348 (5)	C13—H13A	0.9800
C4—C5	1.527 (6)	C13—H13B	0.9800
C4—H4A	0.9900	C13—H13C	0.9800
C4—Sn—C9	130.01 (17)	H5A—C5—H5B	107.8
C4—Sn—S1	108.82 (14)	O1—C6—O2	122.4 (5)
C9—Sn—S1	108.33 (12)	O1—C6—C5	122.3 (4)
C4—Sn—S2	105.78 (12)	O2—C6—C5	115.2 (4)
C9—Sn—S2	107.32 (11)	O2—C7—C8	111.0 (5)
S1—Sn—S2	88.68 (3)	O2—C7—H7A	109.4
C4—Sn—O3	88.46 (13)	C8—C7—H7A	109.4
C9—Sn—O3	72.38 (13)	O2—C7—H7B	109.4
S1—Sn—O3	72.34 (7)	C8—C7—H7B	109.4
S2—Sn—O3	159.35 (7)	H7A—C7—H7B	108.0
C4—Sn—O1	71.72 (15)	C7—C8—H8A	109.5

## supplementary materials

---

C9—Sn—O1	81.87 (14)	C7—C8—H8B	109.5
S1—Sn—O1	163.04 (7)	H8A—C8—H8B	109.5
S2—Sn—O1	75.14 (7)	C7—C8—H8C	109.5
O3—Sn—O1	124.38 (9)	H8A—C8—H8C	109.5
C1—S1—Sn	97.92 (13)	H8B—C8—H8C	109.5
C2—S2—Sn	97.66 (11)	C10—C9—Sn	111.7 (3)
C3—S3—C2	98.14 (18)	C10—C9—H9A	109.3
C3—S4—C1	97.73 (18)	Sn—C9—H9A	109.3
C6—O1—Sn	107.4 (3)	C10—C9—H9B	109.3
C6—O2—C7	115.0 (4)	Sn—C9—H9B	109.3
C11—O3—Sn	106.9 (2)	H9A—C9—H9B	108.0
C11—O4—C12	117.1 (3)	C11—C10—C9	112.7 (4)
C2—C1—S4	116.4 (3)	C11—C10—H10A	109.1
C2—C1—S1	127.1 (3)	C9—C10—H10A	109.1
S4—C1—S1	116.5 (2)	C11—C10—H10B	109.1
C1—C2—S3	115.4 (3)	C9—C10—H10B	109.1
C1—C2—S2	127.9 (3)	H10A—C10—H10B	107.8
S3—C2—S2	116.7 (2)	O3—C11—O4	123.7 (4)
S5—C3—S4	124.2 (2)	O3—C11—C10	123.2 (4)
S5—C3—S3	123.6 (2)	O4—C11—C10	113.1 (4)
S4—C3—S3	112.2 (2)	O4—C12—C13	111.4 (4)
C5—C4—Sn	111.2 (3)	O4—C12—H12A	109.3
C5—C4—H4A	109.4	C13—C12—H12A	109.3
Sn—C4—H4A	109.4	O4—C12—H12B	109.3
C5—C4—H4B	109.4	C13—C12—H12B	109.3
Sn—C4—H4B	109.4	H12A—C12—H12B	108.0
H4A—C4—H4B	108.0	C12—C13—H13A	109.5
C6—C5—C4	112.6 (4)	C12—C13—H13B	109.5
C6—C5—H5A	109.1	H13A—C13—H13B	109.5
C4—C5—H5A	109.1	C12—C13—H13C	109.5
C6—C5—H5B	109.1	H13A—C13—H13C	109.5
C4—C5—H5B	109.1	H13B—C13—H13C	109.5
C4—Sn—S1—C1	99.3 (2)	Sn—S2—C2—S3	174.2 (3)
C9—Sn—S1—C1	-114.8 (2)	C1—S4—C3—S5	-176.2 (3)
S2—Sn—S1—C1	-6.96 (17)	C1—S4—C3—S3	3.4 (3)
O3—Sn—S1—C1	-178.67 (18)	C2—S3—C3—S5	176.1 (3)
O1—Sn—S1—C1	10.3 (3)	C2—S3—C3—S4	-3.5 (3)
C4—Sn—S2—C2	-102.3 (2)	C9—Sn—C4—C5	94.0 (3)
C9—Sn—S2—C2	115.7 (2)	S1—Sn—C4—C5	-129.9 (3)
S1—Sn—S2—C2	6.90 (19)	S2—Sn—C4—C5	-35.8 (3)
O3—Sn—S2—C2	29.8 (3)	O3—Sn—C4—C5	159.3 (3)
O1—Sn—S2—C2	-168.0 (2)	O1—Sn—C4—C5	32.2 (3)
C4—Sn—O1—C6	-24.6 (3)	Sn—C4—C5—C6	-40.7 (5)
C9—Sn—O1—C6	-161.6 (3)	Sn—O1—C6—O2	-168.4 (4)
S1—Sn—O1—C6	70.1 (4)	Sn—O1—C6—C5	9.8 (5)
S2—Sn—O1—C6	88.0 (3)	C7—O2—C6—O1	3.0 (7)
O3—Sn—O1—C6	-99.5 (3)	C7—O2—C6—C5	-175.4 (5)
C4—Sn—O3—C11	-156.3 (3)	C4—C5—C6—O1	17.7 (6)
C9—Sn—O3—C11	-23.1 (3)	C4—C5—C6—O2	-163.9 (4)

S1—Sn—O3—C11	93.4 (3)	C6—O2—C7—C8	164.1 (5)
S2—Sn—O3—C11	69.3 (4)	C4—Sn—C9—C10	102.9 (4)
O1—Sn—O3—C11	-89.7 (3)	S1—Sn—C9—C10	-33.2 (3)
C3—S4—C1—C2	-2.0 (4)	S2—Sn—C9—C10	-127.7 (3)
C3—S4—C1—S1	178.3 (3)	O3—Sn—C9—C10	30.6 (3)
Sn—S1—C1—C2	6.4 (5)	O1—Sn—C9—C10	160.7 (3)
Sn—S1—C1—S4	-173.9 (2)	Sn—C9—C10—C11	-38.3 (4)
S4—C1—C2—S3	-0.2 (5)	Sn—O3—C11—O4	-169.2 (3)
S1—C1—C2—S3	179.5 (3)	Sn—O3—C11—C10	9.5 (5)
S4—C1—C2—S2	-179.9 (3)	C12—O4—C11—O3	3.7 (6)
S1—C1—C2—S2	-0.2 (7)	C12—O4—C11—C10	-175.2 (4)
C3—S3—C2—C1	2.3 (4)	C9—C10—C11—O3	16.6 (6)
C3—S3—C2—S2	-178.0 (3)	C9—C10—C11—O4	-164.6 (4)
Sn—S2—C2—C1	-6.1 (5)	C11—O4—C12—C13	81.2 (5)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C12—H12a $\cdots$ O1 <sup>i</sup>	0.99	2.38	3.338 (6)	164
C7—H7a $\cdots$ O3 <sup>ii</sup>	0.99	2.46	3.450 (7)	178

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



Fig. 2

