

## $\mu$ -Fumarato- $\kappa^2$ O:O'-bis[tris(2-methyl-2-phenylpropyl)tin(IV)]

Xi-Cheng Liu, Ling Yin, Yu-Xi Sun and Lai-Jin Tian\*

Department of Chemistry, Qufu Normal University, Qufu 273165, People's Republic of China

Correspondence e-mail: laijintian@163.com

Received 17 May 2007; accepted 18 May 2007

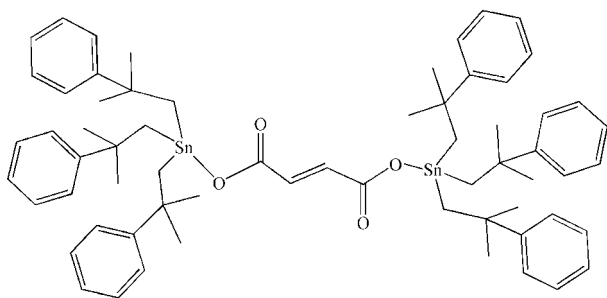
 Key indicators: single-crystal X-ray study;  $T = 295$  K; mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å;  $R$  factor = 0.039;  $wR$  factor = 0.101; data-to-parameter ratio = 21.3.

In the centrosymmetric title compound,  $[\text{Sn}_2(\text{C}_{10}\text{H}_{13})_6(\text{C}_4\text{H}_2\text{O}_4)]$ , the Sn atom adopts a distorted tetrahedral  $\text{SnC}_3\text{O}$  geometry, with a mean Sn—C distance of 2.143 (3) Å and with Sn—O = 2.070 (2) Å. A short Sn...O contact of 3.072 (4) Å is also present.

### Related literature

For related structures, see: Tian *et al.* (2004, 2006); Tian, Sun, Yang & Ng (2005); Tian, Sun, Yang & Yang (2005).

For related literature, see: Chandrasekhar *et al.* (2002); Gielen *et al.* (2005).



### Experimental

#### Crystal data

 $[\text{Sn}_2(\text{C}_{10}\text{H}_{13})_6(\text{C}_4\text{H}_2\text{O}_4)]$   
 $M_r = 1150.66$ 

 Monoclinic,  $P2_1/n$   
 $a = 9.6017$  (9) Å

 $b = 18.6352$  (18) Å  
 $c = 16.5097$  (16) Å  
 $\beta = 91.985$  (1)°  
 $V = 2952.3$  (5) Å<sup>3</sup>  
 $Z = 2$ 

 Mo  $K\alpha$  radiation  
 $\mu = 0.89$  mm<sup>-1</sup>  
 $T = 295$  (2) K  
 $0.22 \times 0.10 \times 0.08$  mm

#### Data collection

 Bruker SMART APEX CCD area-detector diffractometer  
 Absorption correction: multi-scan (SADABS; Bruker, 2002)  
 $T_{\min} = 0.828$ ,  $T_{\max} = 0.932$ 

 23553 measured reflections  
 6102 independent reflections  
 4565 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.048$ 

#### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.039$   
 $wR(F^2) = 0.101$   
 $S = 1.03$   
 6102 reflections

 286 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.67$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.40$  e Å<sup>-3</sup>
**Table 1**

Selected bond lengths (Å).

Sn1—O1	2.070 (2)	Sn1—C3	2.142 (4)
Sn1—C13	2.140 (4)	Sn1—C23	2.145 (3)

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

The authors thank the Science Foundation of Shandong Province and Qufu Normal University for supporting this work.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2424).

### References

- Bruker (2002). SADABS, SAINT and SMART. Bruker AXS Inc., Madison, Wisconsin, USA.
- Chandrasekhar, V., Nagendran, S. & Baskar, V. (2002). *Coord. Chem. Rev.* **235**, 1–52.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Gielen, M., Biesemans, M. & Willem, R. (2005). *Appl. Organomet. Chem.* **19**, 440–450.
- Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.
- Tian, L.-J., Sun, Y.-X. & Ng, S. W. (2004). *Acta Cryst.* **E60**, m1752–m1753.
- Tian, L.-J., Sun, Y.-X., Yang, M. & Ng, S. W. (2005). *Acta Cryst.* **E61**, m74–m75.
- Tian, L.-J., Sun, Y.-X., Yang, M. & Yang, G.-M. (2005). *Acta Cryst.* **E61**, m1346–m1347.
- Tian, L.-J., Yu, F.-Y., Sun, Y.-X. & Liu, X.-C. (2006). *Acta Cryst.* **E62**, m488–m489.

**supplementary materials**

*Acta Cryst.* (2007). E63, m1758 [ doi:10.1107/S1600536807024543 ]

## $\mu$ -Fumarato- $\kappa^2 O:O'$ -bis[tris(2-methyl-2-phenylpropyl)tin(IV)]

X.-C. Liu, L. Yin, Y.-X. Sun and L.-J. Tian

### Comment

The structural chemistry of organotin carboxylates continues to receive attention owing to their biological properties, especially their antitumour activities (Chandrasekhar *et al.*, 2002; Gielen *et al.*, 2005). Recently, we have reported several tris(2-methyl-2-phenylpropyl)tin carboxylates, such as bis[tris(2-methyl-2-phenylpropyl)tin(IV)] 3,4,5,6-tetrafluorophthalate (Tian *et al.*, 2004), bis[tris(2-methyl-2-phenylpropyl)tin(IV)] phthalate (Tian, Sun, Yang & Ng, 2005), tris(2-methyl-2-phenylpropyl)tin pyridine-3-carboxylate (Tian, Sun, Yang & Yang, 2005) and tris(2-methyl-2-phenylpropyl)tin 2-phthalimidoacetate (Tian *et al.*, 2006), which all possess a distorted tetrahedral geometry. In the title compound, (I), tetrahedral coordination is also observed (Fig. 1 & Table 1). The Sn $\cdots$ O2 separation of 3.072 (4)Å indicates there is a weak interaction between these atoms, which distorts the tetrahedral geometry. The three Sn—C distances are almost identical. The Sn—O bond length in (I) is similar to that found in the carboxylate structures mentioned above.

### Experimental

Bis[tris(2-methyl-2-phenylpropyl)tin] oxide (1.05 g, 1 mmol) and fumaric acid (0.12 g, 1 mmol) in toluene (50 ml) were refluxed for 3 h with azeotropic removal of water *via* a Dean-Stark trap. The resulting clear solution was evaporated under reduced pressure. The white solid obtained was purified by recrystallization from methanol, and crystals of (I) were obtained from a chloroform-hexane (1:1, *v/v*) solution by slow evaporation at 298 K (yield 83%, m.p. 422–423 K). Analysis, found: C 66.56, H 6.89%; calculated for C<sub>64</sub>H<sub>80</sub>O<sub>4</sub>Sn<sub>2</sub>: C 66.80, H 7.01%.

### Refinement

The H atoms were placed at calculated positions (C—H = 0.93–0.97 Å) and refined as riding with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  or  $1.5U_{\text{eq}}(\text{methyl C})$ .

### Figures

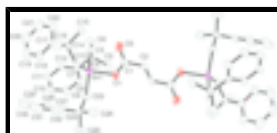


Fig. 1. The structure of (I), with displacement ellipsoids drawn at the 30% probability level. H atoms have been omitted for clarity.

## $\mu$ -Fumarato- $\kappa^2 O,O'$ -bis[tris(2-methyl-2-phenylpropyl)tin(IV)]

### Crystal data

[Sn<sub>2</sub>(C<sub>10</sub>H<sub>13</sub>)<sub>6</sub>(C<sub>4</sub>H<sub>2</sub>O<sub>4</sub>)]

$F_{000} = 1192$

# supplementary materials

---

$M_r = 1150.66$

Monoclinic,  $P2_1/n$

Hall symbol: -P 2yn

$a = 9.6017$  (9) Å

$b = 18.6352$  (18) Å

$c = 16.5097$  (16) Å

$\beta = 91.985$  (1)°

$V = 2952.3$  (5) Å<sup>3</sup>

$Z = 2$

$D_x = 1.294$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 4827 reflections

$\theta = 2.5$ – $22.3$ °

$\mu = 0.89$  mm<sup>-1</sup>

$T = 295$  (2) K

Prism, colourless

$0.22 \times 0.10 \times 0.08$  mm

## Data collection

Bruker SMART APEX CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 295$ (2) K

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan (SADABS; Bruker, 2002)

$T_{\min} = 0.828$ ,  $T_{\max} = 0.932$

23553 measured reflections

6102 independent reflections

4565 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.048$

$\theta_{\max} = 26.5$ °

$\theta_{\min} = 1.7$ °

$h = -12 \rightarrow 12$

$k = -23 \rightarrow 22$

$l = -20 \rightarrow 20$

## Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.039$

$wR(F^2) = 0.101$

$S = 1.03$

6102 reflections

286 parameters

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

$$w = 1/[\sigma^2(F_o^2) + (0.0466P)^2 + 0.84P]$$

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

$(\Delta/\sigma)_{\max} = 0.002$

$\Delta\rho_{\max} = 0.67$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.40$  e Å<sup>-3</sup>

Extinction correction: none

## 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	0.26662 (2)	0.875316 (12)	0.183016 (13)	0.03679 (9)
O1	0.1351 (3)	0.93708 (15)	0.10958 (16)	0.0591 (7)
O2	0.1864 (5)	0.87575 (19)	0.0013 (2)	0.1071 (14)
C1	0.1220 (5)	0.9236 (2)	0.0345 (3)	0.0626 (11)
C2	0.0287 (4)	0.9720 (2)	-0.0139 (2)	0.0552 (10)
H2	0.0110	0.9606	-0.0681	0.066*
C3	0.4650 (4)	0.89136 (19)	0.1303 (2)	0.0457 (9)
H3A	0.5190	0.8477	0.1378	0.055*
H3B	0.4496	0.8981	0.0725	0.055*
C4	0.5541 (4)	0.9550 (2)	0.1633 (2)	0.0474 (9)
C5	0.4787 (5)	1.0247 (2)	0.1402 (3)	0.0760 (14)
H5A	0.3932	1.0276	0.1685	0.114*
H5B	0.4587	1.0253	0.0828	0.114*
H5C	0.5369	1.0648	0.1548	0.114*
C6	0.6948 (4)	0.9532 (3)	0.1223 (3)	0.0793 (14)
H6A	0.7515	0.9924	0.1419	0.119*
H6B	0.6800	0.9575	0.0647	0.119*
H6C	0.7410	0.9086	0.1346	0.119*
C7	0.5821 (3)	0.94903 (13)	0.25515 (11)	0.0463 (9)
C8	0.6025 (3)	1.01064 (11)	0.30152 (17)	0.0678 (12)
H8	0.5989	1.0555	0.2769	0.081*
C9	0.6283 (3)	1.00515 (16)	0.38465 (16)	0.0858 (16)
H9	0.6420	1.0464	0.4157	0.103*
C10	0.6338 (3)	0.9381 (2)	0.42142 (11)	0.0814 (15)
H10	0.6511	0.9344	0.4770	0.098*
C11	0.6134 (3)	0.87645 (15)	0.37504 (16)	0.0726 (13)
H11	0.6171	0.8316	0.3996	0.087*
C12	0.5876 (3)	0.88194 (11)	0.29191 (16)	0.0578 (10)
H12	0.5740	0.8407	0.2609	0.069*
C13	0.1789 (4)	0.7698 (2)	0.1786 (2)	0.0561 (10)
H13A	0.1544	0.7571	0.2333	0.067*
H13B	0.0924	0.7725	0.1465	0.067*
C14	0.2641 (5)	0.7068 (2)	0.1450 (2)	0.0586 (11)
C15	0.2827 (5)	0.7185 (2)	0.0543 (2)	0.0705 (13)
H15A	0.3296	0.7633	0.0461	0.106*
H15B	0.1930	0.7195	0.0268	0.106*
H15C	0.3371	0.6800	0.0332	0.106*
C16	0.1767 (7)	0.6383 (2)	0.1551 (4)	0.0941 (18)
H16A	0.2246	0.5982	0.1325	0.141*
H16B	0.0878	0.6441	0.1275	0.141*
H16C	0.1632	0.6299	0.2117	0.141*
C17	0.4028 (3)	0.69769 (16)	0.19162 (18)	0.0671 (12)

## supplementary materials

---

C18	0.4029 (4)	0.69271 (18)	0.27563 (19)	0.0906 (17)
H18	0.3196	0.6961	0.3025	0.109*
C19	0.5275 (6)	0.6826 (2)	0.31953 (18)	0.128 (3)
H19	0.5276	0.6793	0.3757	0.154*
C20	0.6521 (4)	0.6776 (2)	0.2794 (3)	0.143 (3)
H20	0.7354	0.6708	0.3088	0.172*
C21	0.6520 (3)	0.6825 (2)	0.1954 (3)	0.134 (3)
H21	0.7353	0.6791	0.1686	0.161*
C22	0.5274 (4)	0.69260 (18)	0.15152 (19)	0.0906 (16)
H22	0.5273	0.6959	0.0953	0.109*
C23	0.2213 (4)	0.9367 (2)	0.2888 (2)	0.0487 (9)
H23A	0.3047	0.9374	0.3237	0.058*
H23B	0.2033	0.9857	0.2718	0.058*
C24	0.0987 (4)	0.9124 (2)	0.3405 (2)	0.0580 (11)
C25	0.0768 (5)	0.9691 (3)	0.4075 (3)	0.0903 (17)
H25A	0.0043	0.9533	0.4419	0.135*
H25B	0.0507	1.0140	0.3829	0.135*
H25C	0.1618	0.9751	0.4391	0.135*
C26	-0.0320 (4)	0.9098 (3)	0.2859 (3)	0.0937 (17)
H26A	-0.0236	0.8718	0.2471	0.141*
H26B	-0.0433	0.9547	0.2580	0.141*
H26C	-0.1116	0.9011	0.3182	0.141*
C27	0.1309 (4)	0.84148 (16)	0.38337 (18)	0.0645 (12)
C28	0.2573 (3)	0.8341 (2)	0.4262 (2)	0.0865 (15)
H28	0.3213	0.8715	0.4272	0.104*
C29	0.2879 (5)	0.7707 (3)	0.4675 (2)	0.137 (3)
H29	0.3724	0.7657	0.4962	0.165*
C30	0.1921 (7)	0.71473 (19)	0.4660 (3)	0.161 (4)
H30	0.2126	0.6723	0.4936	0.193*
C31	0.0657 (6)	0.72215 (18)	0.4231 (3)	0.147 (4)
H31	0.0016	0.6847	0.4221	0.177*
C32	0.0351 (4)	0.7855 (2)	0.3818 (2)	0.099 (2)
H32	-0.0494	0.7905	0.3532	0.119*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Sn1	0.04145 (15)	0.03912 (14)	0.02958 (13)	0.00480 (10)	-0.00185 (9)	-0.00045 (10)
O1	0.0569 (16)	0.0752 (19)	0.0443 (16)	0.0170 (14)	-0.0124 (13)	0.0083 (14)
O2	0.154 (4)	0.100 (3)	0.065 (2)	0.063 (3)	-0.035 (2)	-0.0122 (19)
C1	0.070 (3)	0.064 (3)	0.053 (3)	0.012 (2)	-0.013 (2)	0.002 (2)
C2	0.059 (3)	0.064 (3)	0.042 (2)	0.005 (2)	-0.0093 (18)	0.0083 (19)
C3	0.044 (2)	0.053 (2)	0.041 (2)	0.0044 (16)	0.0028 (16)	-0.0011 (16)
C4	0.053 (2)	0.052 (2)	0.038 (2)	-0.0074 (18)	-0.0001 (17)	0.0064 (16)
C5	0.107 (4)	0.056 (3)	0.063 (3)	-0.010 (3)	-0.025 (3)	0.019 (2)
C6	0.063 (3)	0.122 (4)	0.054 (3)	-0.029 (3)	0.012 (2)	0.003 (3)
C7	0.0359 (19)	0.059 (2)	0.045 (2)	-0.0005 (17)	0.0035 (16)	0.0058 (17)
C8	0.083 (3)	0.061 (3)	0.059 (3)	0.001 (2)	-0.006 (2)	-0.004 (2)

C9	0.103 (4)	0.100 (4)	0.052 (3)	0.014 (3)	-0.015 (3)	-0.019 (3)
C10	0.066 (3)	0.137 (5)	0.040 (2)	0.005 (3)	-0.007 (2)	0.009 (3)
C11	0.060 (3)	0.099 (4)	0.058 (3)	-0.006 (3)	-0.011 (2)	0.031 (3)
C12	0.050 (2)	0.066 (3)	0.057 (2)	-0.005 (2)	-0.0041 (19)	0.014 (2)
C13	0.066 (3)	0.050 (2)	0.052 (2)	-0.012 (2)	0.010 (2)	-0.0066 (18)
C14	0.093 (3)	0.039 (2)	0.045 (2)	-0.002 (2)	0.012 (2)	-0.0017 (17)
C15	0.102 (4)	0.062 (3)	0.047 (2)	0.008 (3)	0.003 (2)	-0.013 (2)
C16	0.139 (5)	0.048 (3)	0.096 (4)	-0.025 (3)	0.012 (4)	-0.007 (3)
C17	0.107 (4)	0.038 (2)	0.056 (3)	0.013 (2)	0.005 (3)	-0.0013 (19)
C18	0.138 (5)	0.077 (3)	0.056 (3)	0.012 (3)	-0.007 (3)	0.015 (3)
C19	0.197 (8)	0.087 (4)	0.096 (5)	0.025 (5)	-0.053 (5)	0.010 (4)
C20	0.156 (7)	0.100 (5)	0.169 (8)	0.060 (5)	-0.077 (6)	-0.004 (5)
C21	0.128 (6)	0.117 (6)	0.156 (7)	0.054 (5)	-0.019 (5)	-0.007 (5)
C22	0.104 (4)	0.080 (4)	0.087 (4)	0.037 (3)	-0.004 (3)	0.002 (3)
C23	0.054 (2)	0.053 (2)	0.039 (2)	0.0036 (18)	0.0033 (17)	-0.0132 (17)
C24	0.045 (2)	0.078 (3)	0.052 (2)	-0.002 (2)	0.0084 (18)	-0.026 (2)
C25	0.099 (4)	0.098 (4)	0.076 (3)	0.007 (3)	0.029 (3)	-0.042 (3)
C26	0.040 (2)	0.154 (5)	0.087 (4)	0.010 (3)	0.007 (2)	-0.030 (4)
C27	0.073 (3)	0.075 (3)	0.047 (2)	-0.021 (3)	0.031 (2)	-0.017 (2)
C28	0.093 (4)	0.098 (4)	0.069 (3)	0.007 (3)	0.016 (3)	0.021 (3)
C29	0.183 (8)	0.148 (7)	0.083 (4)	0.035 (6)	0.038 (5)	0.043 (5)
C30	0.288 (13)	0.109 (6)	0.091 (5)	0.019 (7)	0.092 (7)	0.033 (5)
C31	0.238 (11)	0.104 (6)	0.106 (6)	-0.060 (6)	0.099 (7)	-0.021 (5)
C32	0.122 (5)	0.104 (4)	0.075 (4)	-0.042 (4)	0.055 (3)	-0.025 (3)

*Geometric parameters (Å, °)*

Sn1—O1	2.070 (2)	C15—H15B	0.9600
Sn1—C13	2.140 (4)	C15—H15C	0.9600
Sn1—C3	2.142 (4)	C16—H16A	0.9600
Sn1—C23	2.145 (3)	C16—H16B	0.9600
O1—C1	1.267 (5)	C16—H16C	0.9600
O2—C1	1.225 (5)	C17—C18	1.3900
C1—C2	1.485 (5)	C17—C22	1.3900
C2—C2 <sup>i</sup>	1.273 (7)	C18—C19	1.3900
C2—H2	0.9300	C18—H18	0.9300
C3—C4	1.549 (5)	C19—C20	1.3900
C3—H3A	0.9700	C19—H19	0.9300
C3—H3B	0.9700	C20—C21	1.3900
C4—C5	1.529 (5)	C20—H20	0.9300
C4—C6	1.532 (5)	C21—C22	1.3900
C4—C7	1.535 (4)	C21—H21	0.9300
C5—H5A	0.9600	C22—H22	0.9300
C5—H5B	0.9600	C23—C24	1.545 (5)
C5—H5C	0.9600	C23—H23A	0.9700
C6—H6A	0.9600	C23—H23B	0.9700
C6—H6B	0.9600	C24—C26	1.521 (6)
C6—H6C	0.9600	C24—C27	1.526 (5)
C7—C8	1.3900	C24—C25	1.548 (5)

## supplementary materials

---

C7—C12	1.3900	C25—H25A	0.9600
C8—C9	1.3900	C25—H25B	0.9600
C8—H8	0.9300	C25—H25C	0.9600
C9—C10	1.3900	C26—H26A	0.9600
C9—H9	0.9300	C26—H26B	0.9600
C10—C11	1.3900	C26—H26C	0.9600
C10—H10	0.9300	C27—C28	1.3900
C11—C12	1.3900	C27—C32	1.3900
C11—H11	0.9300	C28—C29	1.3900
C12—H12	0.9300	C28—H28	0.9300
C13—C14	1.544 (5)	C29—C30	1.3900
C13—H13A	0.9700	C29—H29	0.9300
C13—H13B	0.9700	C30—C31	1.3900
C14—C17	1.525 (5)	C30—H30	0.9300
C14—C15	1.529 (5)	C31—C32	1.3900
C14—C16	1.540 (6)	C31—H31	0.9300
C15—H15A	0.9600	C32—H32	0.9300
O1—Sn1—C13	105.04 (14)	C14—C15—H15C	109.5
O1—Sn1—C3	102.57 (13)	H15A—C15—H15C	109.5
C13—Sn1—C3	117.96 (14)	H15B—C15—H15C	109.5
O1—Sn1—C23	92.50 (13)	C14—C16—H16A	109.5
C13—Sn1—C23	115.29 (14)	C14—C16—H16B	109.5
C3—Sn1—C23	117.68 (14)	H16A—C16—H16B	109.5
C1—O1—Sn1	120.0 (3)	C14—C16—H16C	109.5
O2—C1—O1	123.2 (4)	H16A—C16—H16C	109.5
O2—C1—C2	120.4 (4)	H16B—C16—H16C	109.5
O1—C1—C2	116.3 (4)	C18—C17—C22	120.0
C2 <sup>i</sup> —C2—C1	124.4 (5)	C18—C17—C14	118.8 (3)
C2 <sup>i</sup> —C2—H2	117.8	C22—C17—C14	121.2 (3)
C1—C2—H2	117.8	C19—C18—C17	120.0
C4—C3—Sn1	116.9 (2)	C19—C18—H18	120.0
C4—C3—H3A	108.1	C17—C18—H18	120.0
Sn1—C3—H3A	108.1	C18—C19—C20	120.0
C4—C3—H3B	108.1	C18—C19—H19	120.0
Sn1—C3—H3B	108.1	C20—C19—H19	120.0
H3A—C3—H3B	107.3	C21—C20—C19	120.0
C5—C4—C6	109.0 (3)	C21—C20—H20	120.0
C5—C4—C7	111.9 (3)	C19—C20—H20	120.0
C6—C4—C7	107.9 (3)	C20—C21—C22	120.0
C5—C4—C3	108.2 (3)	C20—C21—H21	120.0
C6—C4—C3	108.2 (3)	C22—C21—H21	120.0
C7—C4—C3	111.6 (3)	C21—C22—C17	120.0
C4—C5—H5A	109.5	C21—C22—H22	120.0
C4—C5—H5B	109.5	C17—C22—H22	120.0
H5A—C5—H5B	109.5	C24—C23—Sn1	118.3 (2)
C4—C5—H5C	109.5	C24—C23—H23A	107.7
H5A—C5—H5C	109.5	Sn1—C23—H23A	107.7
H5B—C5—H5C	109.5	C24—C23—H23B	107.7



C4—C6—H6A	109.5	Sn1—C23—H23B	107.7
C4—C6—H6B	109.5	H23A—C23—H23B	107.1
H6A—C6—H6B	109.5	C26—C24—C27	113.3 (4)
C4—C6—H6C	109.5	C26—C24—C23	108.1 (4)
H6A—C6—H6C	109.5	C27—C24—C23	111.3 (3)
H6B—C6—H6C	109.5	C26—C24—C25	108.4 (4)
C8—C7—C12	120.0	C27—C24—C25	106.9 (3)
C8—C7—C4	120.1 (2)	C23—C24—C25	108.7 (3)
C12—C7—C4	119.9 (2)	C24—C25—H25A	109.5
C7—C8—C9	120.0	C24—C25—H25B	109.5
C7—C8—H8	120.0	H25A—C25—H25B	109.5
C9—C8—H8	120.0	C24—C25—H25C	109.5
C8—C9—C10	120.0	H25A—C25—H25C	109.5
C8—C9—H9	120.0	H25B—C25—H25C	109.5
C10—C9—H9	120.0	C24—C26—H26A	109.5
C11—C10—C9	120.0	C24—C26—H26B	109.5
C11—C10—H10	120.0	H26A—C26—H26B	109.5
C9—C10—H10	120.0	C24—C26—H26C	109.5
C12—C11—C10	120.0	H26A—C26—H26C	109.5
C12—C11—H11	120.0	H26B—C26—H26C	109.5
C10—C11—H11	120.0	C28—C27—C32	120.0
C11—C12—C7	120.0	C28—C27—C24	118.7 (3)
C11—C12—H12	120.0	C32—C27—C24	121.2 (3)
C7—C12—H12	120.0	C29—C28—C27	120.0
C14—C13—Sn1	119.9 (3)	C29—C28—H28	120.0
C14—C13—H13A	107.4	C27—C28—H28	120.0
Sn1—C13—H13A	107.4	C30—C29—C28	120.0
C14—C13—H13B	107.4	C30—C29—H29	120.0
Sn1—C13—H13B	107.4	C28—C29—H29	120.0
H13A—C13—H13B	106.9	C29—C30—C31	120.0
C17—C14—C15	112.4 (4)	C29—C30—H30	120.0
C17—C14—C16	108.8 (4)	C31—C30—H30	120.0
C15—C14—C16	107.9 (4)	C30—C31—C32	120.0
C17—C14—C13	111.6 (3)	C30—C31—H31	120.0
C15—C14—C13	108.9 (3)	C32—C31—H31	120.0
C16—C14—C13	107.1 (4)	C31—C32—C27	120.0
C14—C15—H15A	109.5	C31—C32—H32	120.0
C14—C15—H15B	109.5	C27—C32—H32	120.0
H15A—C15—H15B	109.5		
C13—Sn1—O1—C1	-63.6 (3)	C16—C14—C17—C18	-66.0 (4)
C3—Sn1—O1—C1	60.3 (3)	C13—C14—C17—C18	51.9 (4)
C23—Sn1—O1—C1	179.4 (3)	C15—C14—C17—C22	-7.0 (4)
Sn1—O1—C1—O2	-1.6 (6)	C16—C14—C17—C22	112.4 (3)
Sn1—O1—C1—C2	-178.3 (3)	C13—C14—C17—C22	-129.6 (3)
O2—C1—C2—C2 <sup>i</sup>	-171.2 (6)	C22—C17—C18—C19	0.0
O1—C1—C2—C2 <sup>i</sup>	5.6 (8)	C14—C17—C18—C19	178.4 (3)
O1—Sn1—C3—C4	90.0 (3)	C17—C18—C19—C20	0.0
C13—Sn1—C3—C4	-155.2 (3)	C18—C19—C20—C21	0.0

## supplementary materials

---

C23—Sn1—C3—C4	-9.6 (3)	C19—C20—C21—C22	0.0
Sn1—C3—C4—C5	-66.3 (4)	C20—C21—C22—C17	0.0
Sn1—C3—C4—C6	175.8 (3)	C18—C17—C22—C21	0.0
Sn1—C3—C4—C7	57.2 (4)	C14—C17—C22—C21	-178.4 (3)
C5—C4—C7—C8	-30.0 (4)	O1—Sn1—C23—C24	90.3 (3)
C6—C4—C7—C8	89.8 (3)	C13—Sn1—C23—C24	-17.6 (4)
C3—C4—C7—C8	-151.4 (2)	C3—Sn1—C23—C24	-164.1 (3)
C5—C4—C7—C12	150.1 (3)	Sn1—C23—C24—C26	-56.6 (4)
C6—C4—C7—C12	-90.0 (3)	Sn1—C23—C24—C27	68.5 (4)
C3—C4—C7—C12	28.8 (4)	Sn1—C23—C24—C25	-174.0 (3)
C12—C7—C8—C9	0.0	C26—C24—C27—C28	171.4 (3)
C4—C7—C8—C9	-179.8 (3)	C23—C24—C27—C28	49.3 (4)
C7—C8—C9—C10	0.0	C25—C24—C27—C28	-69.3 (4)
C8—C9—C10—C11	0.0	C26—C24—C27—C32	-10.2 (4)
C9—C10—C11—C12	0.0	C23—C24—C27—C32	-132.4 (3)
C10—C11—C12—C7	0.0	C25—C24—C27—C32	109.1 (3)
C8—C7—C12—C11	0.0	C32—C27—C28—C29	0.0
C4—C7—C12—C11	179.8 (3)	C24—C27—C28—C29	178.4 (3)
O1—Sn1—C13—C14	118.8 (3)	C27—C28—C29—C30	0.0
C3—Sn1—C13—C14	5.3 (4)	C28—C29—C30—C31	0.0
C23—Sn1—C13—C14	-141.0 (3)	C29—C30—C31—C32	0.0
Sn1—C13—C14—C17	57.4 (4)	C30—C31—C32—C27	0.0
Sn1—C13—C14—C15	-67.2 (4)	C28—C27—C32—C31	0.0
Sn1—C13—C14—C16	176.4 (3)	C24—C27—C32—C31	-178.4 (3)
C15—C14—C17—C18	174.6 (3)		

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

