

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

catena-Poly[[trimethyltin(IV)]- $\mu$ -2,4,6-trichlorobenzoato]

Liyuan Wen, Handong Yin,\* Wenkuan Li and Daqi Wang

College of Chemistry and Chemical Engineering, Liaocheng University, Shandong 252059, People's Republic of China

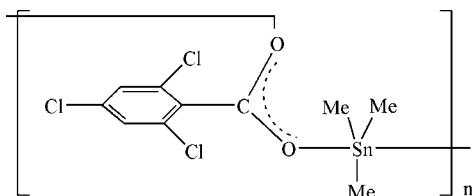
Correspondence e-mail: handongyin@163.com

Received 15 November 2008; accepted 3 December 2008

Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å;  $R$  factor = 0.028;  $wR$  factor = 0.085; data-to-parameter ratio = 17.0.

In the title compound,  $[\text{Sn}(\text{CH}_3)_3(\text{C}_7\text{H}_2\text{Cl}_3\text{O}_2)]_n$ , the tin(IV) atom exhibits a slightly distorted trigonal-bipyramidal geometry with two O atoms of symmetry-related carboxylate groups in the axial positions and three methyl groups in the equatorial positions. In the crystal structure, the metal atoms are linked by carboxylate bridges into polymeric chains extending along the  $b$  axis.

## Related literature

For related structures, see: Wang *et al.* (2007); Ma *et al.* (2006).

## Experimental

## Crystal data

 $[\text{Sn}(\text{CH}_3)_3(\text{C}_7\text{H}_2\text{Cl}_3\text{O}_2)]$   
 $M_r = 388.25$ 

 Monoclinic,  $P2_1/c$   
 $a = 9.8457$  (10) Å

 $b = 9.6891$  (9) Å  
 $c = 15.3028$  (19) Å  
 $\beta = 106.761$  (1)°  
 $V = 1397.8$  (3) Å<sup>3</sup>  
 $Z = 4$ 

 Mo  $K\alpha$  radiation  
 $\mu = 2.38$  mm<sup>-1</sup>  
 $T = 298$  (2) K  
 $0.42 \times 0.18 \times 0.08$  mm

## Data collection

 Bruker SMART CCD area-detector diffractometer  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.434$ ,  $T_{\max} = 0.832$ 

 6983 measured reflections  
 2469 independent reflections  
 1996 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.025$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.028$   
 $wR(F^2) = 0.085$   
 $S = 1.01$   
 2469 reflections

 145 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.68$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.37$  e Å<sup>-3</sup>

Table 1

Selected bond lengths (Å).

Sn1—C9	2.107 (5)	Sn1—O1	2.212 (3)
Sn1—C10	2.116 (5)	Sn1—O2 <sup>i</sup>	2.467 (3)
Sn1—C8	2.123 (4)		

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

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

We acknowledge the National Natural Foundation of China (grant No. 20771053), and the Natural Science Foundation of Shandong Province (2005ZX09) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2273).

## References

- Ma, C., Li, J., Zhang, R. & Wang, D. (2006). *J. Organomet. Chem.*, **691**, 1713–1721.  
 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.  
 Wang, H., Yin, H. & Wang, D. (2007). *Acta Cryst.* **E63**, m2955.

**supplementary materials**

*Acta Cryst.* (2009). E65, m30 [ doi:10.1107/S1600536808040798 ]

### **catena-Poly[[trimethyltin(IV)]- $\mu$ -2,4,6-trichlorobenzoato]**

**L. Wen, H. Yin, W. Li and D. Wang**

#### **Comment**

Organoantimony(IV) derivatives have recently attracted considerable attention due to the significant antimicrobial properties (Wang *et al.*, 2007). As a part of our ongoing investigations in this field, we have synthesized the title compound and present its crystal structure here.

In the title compound (Fig. 1), the Sn—O bond distances (Table 1) are comparable to those found in related organotin carboxylates (Ma *et al.*, 2006). The Sn atom assumes a slightly distorted trigonal-bipyramidal coordination geometry, provided by two O atoms of symmetry related carboxylate groups at the axial positions and three methyl groups at the equatorial positions. In the crystal structure, the metal atoms are linked by carboxylate bridges into polymeric chains extending along the *b* axis (Fig. 2).

#### **Experimental**

The reaction was carried out under nitrogen atmosphere. 2,4,6-Trichlorobenzoic acid (1 mmol) and sodium ethoxide (1.2 mmol) were added to a stirred solution of benzene (30 ml) in a Schlenk flask and stirred for 0.5 h. Trimethyltin chloride (1 mmol) was then added and the reaction mixture was stirred for 12 h at room temperature. The resulting clear solution was evaporated under vacuum. Colourless crystals suitable for X-ray analysis were obtained by slow evaporation of a dichloromethane/methanol (1:1 v/v) solution (yield 83%. m. p. 403K). Anal. Calcd (%) for C<sub>10</sub>H<sub>11</sub>Cl<sub>3</sub>O<sub>2</sub>Sn: C, 30.94; H, 2.86; O, 8.24; Sn, 30.58; Found (%): C, 30.89; H, 2.90; O, 8.31; Sn, 30.62.

#### **Refinement**

H atoms were positioned geometrically, with methyl C—H distances of 0.96 Å and aromatic C—H distances of 0.93 Å, and refined as riding on their parent atoms, with  $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$  or  $1.5 U_{\text{eq}}(\text{C})$  for the methyl groups.

#### **Figures**

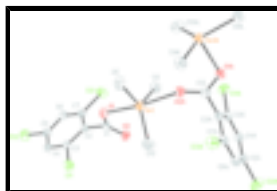


Fig. 1. The molecular structure of the compound, showing 50% probability displacement ellipsoids. H atoms are omitted for clarity. Symmetry code: (A) =  $-x + 1, y + 1/2, -z + 1/2$ .

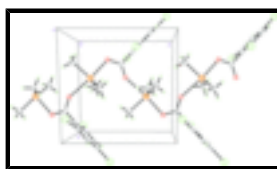


Fig. 2. View of the one-dimensional chain structure extending along the *b* axis.

# supplementary materials

---

## catena-Poly[[trimethyltin(IV)]- $\mu$ -2,4,6-trichlorobenzoato]

### Crystal data

[Sn(CH <sub>3</sub> ) <sub>3</sub> (C <sub>7</sub> H <sub>2</sub> Cl <sub>3</sub> O <sub>2</sub> )]	$F_{000} = 752$
$M_r = 388.25$	$D_x = 1.845 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
Hall symbol: -P 2ybc	$\lambda = 0.71073 \text{ \AA}$
$a = 9.8457 (10) \text{ \AA}$	Cell parameters from 3337 reflections
$b = 9.6891 (9) \text{ \AA}$	$\theta = 2.5\text{--}27.6^\circ$
$c = 15.3028 (19) \text{ \AA}$	$\mu = 2.38 \text{ mm}^{-1}$
$\beta = 106.7610 (10)^\circ$	$T = 298 (2) \text{ K}$
$V = 1397.8 (3) \text{ \AA}^3$	Block, colourless
$Z = 4$	$0.42 \times 0.18 \times 0.08 \text{ mm}$

### Data collection

Bruker SMART CCD area-detector diffractometer	2469 independent reflections
Radiation source: fine-focus sealed tube	1996 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.025$
$T = 298(2) \text{ K}$	$\theta_{\text{max}} = 25.0^\circ$
$\varphi$ and $\omega$ scans	$\theta_{\text{min}} = 2.2^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -9 \rightarrow 11$
$T_{\text{min}} = 0.434$ , $T_{\text{max}} = 0.832$	$k = -10 \rightarrow 11$
6983 measured reflections	$l = -18 \rightarrow 15$

### Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.028$	H-atom parameters constrained
$wR(F^2) = 0.085$	$w = 1/[\sigma^2(F_o^2) + (0.048P)^2 + 1.1107P]$
$S = 1.01$	where $P = (F_o^2 + 2F_c^2)/3$
2469 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
145 parameters	$\Delta\rho_{\text{max}} = 0.68 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.37 \text{ e \AA}^{-3}$
	Extinction correction: none

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

$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
-----	-----	-----	----------------------------------

Sn1	0.41662 (3)	0.26723 (3)	0.24850 (2)	0.03954 (13)
Cl1	0.13395 (13)	-0.14643 (14)	0.30795 (8)	0.0553 (3)
Cl2	-0.17245 (14)	-0.41671 (14)	0.00986 (9)	0.0685 (4)
Cl3	0.21742 (14)	-0.04603 (14)	-0.01909 (8)	0.0589 (3)
O1	0.2441 (3)	0.1152 (3)	0.1981 (2)	0.0436 (7)
O2	0.4091 (3)	-0.0438 (3)	0.2076 (2)	0.0483 (8)
C1	0.2834 (4)	-0.0058 (4)	0.1860 (3)	0.0368 (9)
C2	0.1682 (4)	-0.1073 (4)	0.1414 (3)	0.0357 (9)
C3	0.0947 (4)	-0.1793 (4)	0.1922 (3)	0.0379 (9)
C4	-0.0092 (5)	-0.2761 (4)	0.1529 (3)	0.0416 (10)
H4	-0.0557	-0.3248	0.1881	0.050*
C5	-0.0410 (5)	-0.2974 (4)	0.0609 (3)	0.0429 (11)
C6	0.0256 (5)	-0.2269 (4)	0.0067 (3)	0.0449 (11)
H6	0.0004	-0.2414	-0.0560	0.054*
C7	0.1307 (4)	-0.1341 (4)	0.0483 (3)	0.0396 (10)
C8	0.2673 (5)	0.4245 (5)	0.2502 (4)	0.0582 (13)
H8A	0.3162	0.5044	0.2809	0.087*
H8B	0.2023	0.3917	0.2818	0.087*
H8C	0.2158	0.4484	0.1887	0.087*
C9	0.4924 (6)	0.2466 (5)	0.1337 (3)	0.0510 (12)
H9A	0.5867	0.2092	0.1523	0.077*
H9B	0.4938	0.3355	0.1062	0.077*
H9C	0.4312	0.1857	0.0902	0.077*
C10	0.5129 (6)	0.1758 (6)	0.3770 (3)	0.0617 (14)
H10A	0.6022	0.1359	0.3770	0.093*
H10B	0.4520	0.1051	0.3886	0.093*
H10C	0.5283	0.2449	0.4238	0.093*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Sn1	0.03773 (19)	0.0345 (2)	0.0475 (2)	-0.00090 (12)	0.01410 (14)	-0.00256 (13)
Cl1	0.0537 (7)	0.0745 (9)	0.0398 (7)	-0.0114 (6)	0.0167 (5)	-0.0052 (6)
Cl2	0.0641 (8)	0.0599 (8)	0.0692 (9)	-0.0214 (7)	-0.0003 (6)	-0.0076 (7)
Cl3	0.0717 (8)	0.0616 (8)	0.0511 (7)	-0.0064 (6)	0.0300 (6)	0.0062 (6)
O1	0.0357 (15)	0.0308 (16)	0.066 (2)	-0.0010 (12)	0.0176 (14)	-0.0100 (14)
O2	0.0367 (17)	0.0375 (17)	0.068 (2)	0.0041 (13)	0.0106 (14)	0.0015 (15)
C1	0.038 (2)	0.034 (2)	0.040 (2)	-0.0021 (18)	0.0155 (19)	0.0017 (18)
C2	0.035 (2)	0.028 (2)	0.042 (2)	0.0022 (17)	0.0089 (18)	-0.0017 (18)
C3	0.040 (2)	0.037 (2)	0.038 (2)	-0.0023 (19)	0.0133 (19)	-0.0009 (19)
C4	0.038 (2)	0.036 (2)	0.051 (3)	-0.0047 (18)	0.012 (2)	0.003 (2)
C5	0.039 (2)	0.033 (2)	0.051 (3)	-0.0032 (19)	0.005 (2)	-0.007 (2)
C6	0.049 (3)	0.045 (3)	0.038 (3)	0.004 (2)	0.009 (2)	-0.005 (2)
C7	0.041 (2)	0.035 (2)	0.045 (3)	0.0037 (18)	0.0144 (19)	0.0031 (19)
C8	0.045 (3)	0.045 (3)	0.086 (4)	-0.004 (2)	0.022 (3)	-0.019 (3)
C9	0.055 (3)	0.052 (3)	0.051 (3)	-0.002 (2)	0.023 (2)	0.001 (2)
C10	0.078 (4)	0.056 (3)	0.047 (3)	-0.014 (3)	0.011 (3)	0.004 (2)

## supplementary materials

---

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

Sn1—C9	2.107 (5)	C4—C5	1.366 (6)
Sn1—C10	2.116 (5)	C4—H4	0.9300
Sn1—C8	2.123 (4)	C5—C6	1.377 (7)
Sn1—O1	2.212 (3)	C6—C7	1.380 (6)
Sn1—O2 <sup>i</sup>	2.467 (3)	C6—H6	0.9300
C11—C3	1.730 (4)	C8—H8A	0.9600
C12—C5	1.744 (4)	C8—H8B	0.9600
C13—C7	1.740 (4)	C8—H8C	0.9600
O1—C1	1.265 (5)	C9—H9A	0.9600
O2—C1	1.241 (5)	C9—H9B	0.9600
O2—Sn1 <sup>ii</sup>	2.467 (3)	C9—H9C	0.9600
C1—C2	1.508 (5)	C10—H10A	0.9600
C2—C7	1.390 (6)	C10—H10B	0.9600
C2—C3	1.392 (6)	C10—H10C	0.9600
C3—C4	1.390 (6)		
C9—Sn1—C10	124.3 (2)	C6—C5—C12	118.7 (4)
C9—Sn1—C8	119.4 (2)	C5—C6—C7	118.0 (4)
C10—Sn1—C8	114.6 (2)	C5—C6—H6	121.0
C9—Sn1—O1	93.83 (16)	C7—C6—H6	121.0
C10—Sn1—O1	97.96 (16)	C6—C7—C2	122.4 (4)
C8—Sn1—O1	91.00 (15)	C6—C7—C13	118.5 (4)
C9—Sn1—O2 <sup>i</sup>	84.92 (15)	C2—C7—C13	119.1 (3)
C10—Sn1—O2 <sup>i</sup>	88.16 (16)	Sn1—C8—H8A	109.5
C8—Sn1—O2 <sup>i</sup>	83.90 (15)	Sn1—C8—H8B	109.5
O1—Sn1—O2 <sup>i</sup>	173.28 (10)	H8A—C8—H8B	109.5
C1—O1—Sn1	115.6 (2)	Sn1—C8—H8C	109.5
C1—O2—Sn1 <sup>ii</sup>	148.7 (3)	H8A—C8—H8C	109.5
O2—C1—O1	124.0 (4)	H8B—C8—H8C	109.5
O2—C1—C2	119.3 (4)	Sn1—C9—H9A	109.5
O1—C1—C2	116.6 (3)	Sn1—C9—H9B	109.5
C7—C2—C3	116.9 (4)	H9A—C9—H9B	109.5
C7—C2—C1	121.9 (4)	Sn1—C9—H9C	109.5
C3—C2—C1	121.2 (4)	H9A—C9—H9C	109.5
C4—C3—C2	122.1 (4)	H9B—C9—H9C	109.5
C4—C3—C11	119.1 (3)	Sn1—C10—H10A	109.5
C2—C3—C11	118.7 (3)	Sn1—C10—H10B	109.5
C5—C4—C3	118.0 (4)	H10A—C10—H10B	109.5
C5—C4—H4	121.0	Sn1—C10—H10C	109.5
C3—C4—H4	121.0	H10A—C10—H10C	109.5
C4—C5—C6	122.6 (4)	H10B—C10—H10C	109.5
C4—C5—C12	118.8 (4)		
C9—Sn1—O1—C1	61.1 (3)	C1—C2—C3—C11	2.0 (5)
C10—Sn1—O1—C1	-64.4 (3)	C2—C3—C4—C5	-1.5 (7)
C8—Sn1—O1—C1	-179.3 (3)	C11—C3—C4—C5	178.2 (3)

Sn1 <sup>ii</sup> —O2—C1—O1	154.5 (4)	C3—C4—C5—C6	-0.1 (7)
Sn1 <sup>ii</sup> —O2—C1—C2	-26.3 (8)	C3—C4—C5—C12	-179.2 (3)
Sn1—O1—C1—O2	6.1 (5)	C4—C5—C6—C7	1.7 (7)
Sn1—O1—C1—C2	-173.0 (3)	C12—C5—C6—C7	-179.2 (3)
O2—C1—C2—C7	-82.9 (5)	C5—C6—C7—C2	-1.8 (7)
O1—C1—C2—C7	96.3 (5)	C5—C6—C7—C13	179.1 (3)
O2—C1—C2—C3	96.8 (5)	C3—C2—C7—C6	0.2 (6)
O1—C1—C2—C3	-84.0 (5)	C1—C2—C7—C6	179.9 (4)
C7—C2—C3—C4	1.5 (6)	C3—C2—C7—C13	179.3 (3)
C1—C2—C3—C4	-178.2 (4)	C1—C2—C7—C13	-1.0 (5)
C7—C2—C3—C11	-178.3 (3)		

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



Fig. 2

