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Poly[bis(trimethyltin)-μ-glutarato]

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.006 Å
R factor = 0.031
wR factor = 0.062
Data-to-parameter ratio = 29.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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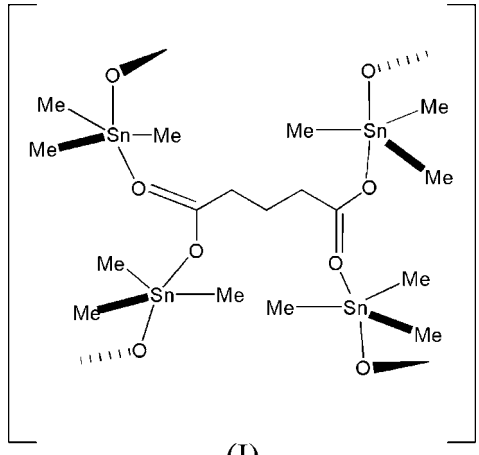
The structure of $[SnMe_3O_2C(CH_2)_3CO_2SnMe_3]_n$ or $\{[Sn_2(CH_3)_6(C_5H_6O_4)]\}_n$ consists of an infinite layer containing pentacoordinate Sn atoms. There are two formula units, A and B, in the asymmetric unit. Glutarate acts as a tetradentate bridging ligand, each glutarate being linked to four different Sn atoms, generating two-dimensional sheets. One ligand links the Sn atoms of unit A, the other the Sn atoms of unit B. The sheets are parallel to each other.

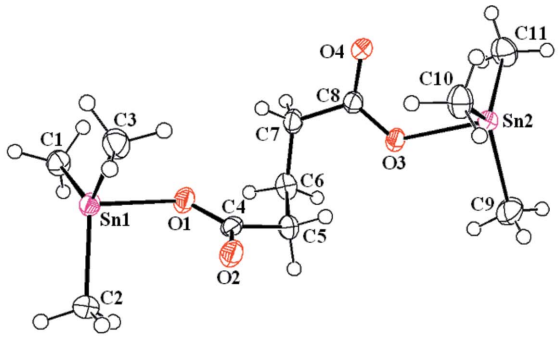
Comment

Crystal structures of $[SnR_3]_2[O_2C(CH_2)CO_2]$ (R = Me and Ph) and $[SnPh_3]_2[O_2C(CH_2)_3CO_2]$ have been reported (Yin *et al.*, 2002; Cissé *et al.*, 2003). In addition to their interest as novel organotin species, bactericidal and fungicidal activity of triorganotin compounds are quite well known (Dutrecq *et al.*, 1992; Moens *et al.*, 1992). Such species are also screened for their antitumour activity and several of them are found to be active *in vitro* (Gielen, 2003, 2005). As a continuation of our interest in triaryltin derivatives (Diassé-Sarr *et al.*, 1997, 2004; Diop *et al.*, 2002), the X-ray crystallographic analysis of the title compound, (I) (Fig. 1), has been carried out and the results are presented here.

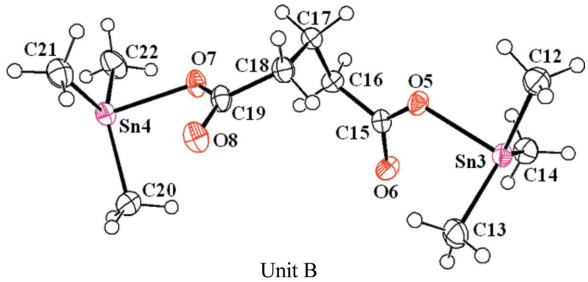


(I)

Each Sn atom is five-coordinate and adopts trigonal–bipyramidal *trans*-O_2SnC_3 geometry. There are two formula units in the asymmetric unit. The O—Sn—O bond angles are in the range 171.32 (10)–173.34 (10)° and are close to the ideal value, while C—Sn—C angles within the equatorial plane of the Sn atoms cover the range 117.35 (17)–123.25 (18)° and confirm the near-planarity of the SnC_3 groups revealed by the IR data. Each Sn atom has short [Sn1—O1 = 2.214 (3) Å] and long [Sn1—O2^i = 2.346 (3) Å; symmetry code as in Table 1]



Unit A



Unit B

Figure 1
The structures of the two formula units in the asymmetric unit of (I), with ellipsoids drawn at the 50% probability level. H atoms have been omitted.

Sn—O bond lengths, which correspond to two C—O bond lengths. The longer C—O bond [C4—O1= 1.276 (5) Å] corresponds to the stronger Sn–O bond and the shorter C—O bond [C4—O2 = 1.244 (5) Å] to the longer (weaker) Sn—O bond (Table 1). In (I), the Sn—O bonds are slightly longer than those reported for the triphenyltin analogue (Yin *et al.*, 2002). The glutarate acts as a tetradentate bridging ligand, each glutarate being linked to four different Sn atoms, generating two-dimensional sheets. A first ligand links Sn1 and Sn2, another one Sn3 and Sn4. The sheets are parallel to each other (Fig. 2). The network incorporates 24-atom rectangular rings with approximate dimensions 9.9 × 6.7 Å measured between Sn atoms on opposite edges of the rectangle.

Experimental

Me_4NOH (10%), $HCO_2(CH_2)_3CO_2H$ and $SnMe_3Cl$ were obtained from Aldrich and were used without further purification. An ethanol solution (20 ml) containing $[Me_4N]_2[CO_2(CH_2)_3CO_2]\cdot 5H_2O$ (0.37 g, 1 mmol) [obtained from a solution of Me_4NOH (20% in water) and $HCO_2(CH_2)_3CO_2H$ in 1:2 ratio] and $SnMe_3Cl$ (0.20 g, 1 mmol) was stirred at room temperature for more than 2 h to give a solution from which colourless crystals were formed after slow solvent evaporation (yield 80%; m.p. 391 K).

Crystal data

$[Sn_2(CH_3)_6(C_5H_6O_4)]$
M_r = 457.68
Orthorhombic, $P2_12_12_1$
a = 10.0053 (1) Å
b = 13.1391 (2) Å
c = 25.9006 (5) Å
V = 3404.83 (9) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 2.94 mm^{-1}
T = 150 (2) K
0.25 × 0.25 × 0.15 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.502, T_{max} = 0.641
42835 measured reflections
9389 independent reflections
8432 reflections with $I > 2\sigma(I)$
R_{int} = 0.058

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.062
S = 1.04
9389 reflections
320 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.72 e Å^{-3}
$\Delta\rho_{min}$ = −1.11 e Å^{-3}
Absolute structure: Flack (1983), 3866 Friedel pairs
Flack parameter: 0.50 (2)

Table 1
Selected geometric parameters (Å, °).

Sn1—O1	2.214 (3)	O2—C4	1.244 (5)
Sn1—O2^{i}	2.346 (3)	O3—C8	1.270 (5)
Sn2—O3	2.201 (3)	O4—C8	1.258 (5)
Sn2—O4^{ii}	2.333 (3)	O5—C15	1.276 (5)
Sn3—O5	2.197 (3)	O6—C15	1.243 (5)
Sn3—O8^{iii}	2.343 (3)	O7—C19	1.268 (5)
Sn4—O7	2.211 (3)	O8—C19	1.255 (5)
O1—C4	1.276 (5)		
C1—Sn1—C2	117.35 (17)	C10—Sn2—O3	94.60 (14)
C1—Sn1—C3	119.91 (18)	C10—Sn2—O4^{ii}	88.79 (14)
C3—Sn1—C2	122.34 (18)	C11—Sn2—O3	95.74 (15)
C10—Sn2—C9	118.3 (2)	C11—Sn2—O4^{ii}	88.60 (14)
C11—Sn2—C10	122.02 (19)	C12—Sn3—O5	88.33 (14)
C11—Sn2—C9	119.1 (2)	C12—Sn3—O8^{iii}	85.05 (14)
C13—Sn3—C12	119.36 (19)	C13—Sn3—O5	94.11 (14)
C14—Sn3—C13	123.19 (18)	C13—Sn3—O8^{iii}	88.54 (14)
C14—Sn3—C12	117.01 (19)	C14—Sn3—O5	93.89 (14)
C20—Sn4—C21	123.25 (18)	C14—Sn3—O8^{iii}	89.75 (14)
C22—Sn4—C20	118.79 (19)	C20—Sn4—O6^{iv}	87.64 (14)
C22—Sn4—C21	117.4 (2)	C21—Sn4—O6^{iv}	89.90 (14)
C1—Sn1—O1	86.64 (13)	C20—Sn4—O7	95.28 (14)
C1—Sn1—O2^{i}	85.01 (13)	C21—Sn4—O7	94.41 (14)
C2—Sn1—O1	95.15 (14)	C22—Sn4—O6^{iv}	84.66 (14)
C2—Sn1—O2^{i}	90.80 (14)	C22—Sn4—O7	87.70 (14)
C3—Sn1—O1	94.33 (14)	O1—Sn1—O2^{i}	171.32 (10)
C3—Sn1—O2^{i}	87.79 (15)	O3—Sn2—O4^{ii}	171.96 (10)
C9—Sn2—O3	87.29 (15)	O5—Sn3—O8^{iii}	173.34 (10)
C9—Sn2—O4^{ii}	84.68 (15)	O7—Sn4—O6^{iv}	172.31 (10)

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{3}{2}, -z+2$; (ii) $x-\frac{1}{2}, -y+\frac{1}{2}, -z+2$; (iii) $-x-1, y+\frac{1}{2}, -z+\frac{3}{2}$; (iv) $-x, y-\frac{1}{2}, -z+\frac{3}{2}$.

The structure was refined as an inversion twin, with components in the ratio 1:1. All H atoms were geometrically located in ideal positions and refined using a riding model, with C—H = 0.98 Å for methyl H atoms and C—H = 0.99 Å for methylene H atoms, and with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and U_{iso}(H) = 1.2U_{eq}(C) for methylene H atoms. The deepest hole in the final difference map is 0.78 Å from Sn4.

Data collection: *COLLECT* (Nonius, 1997); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1998); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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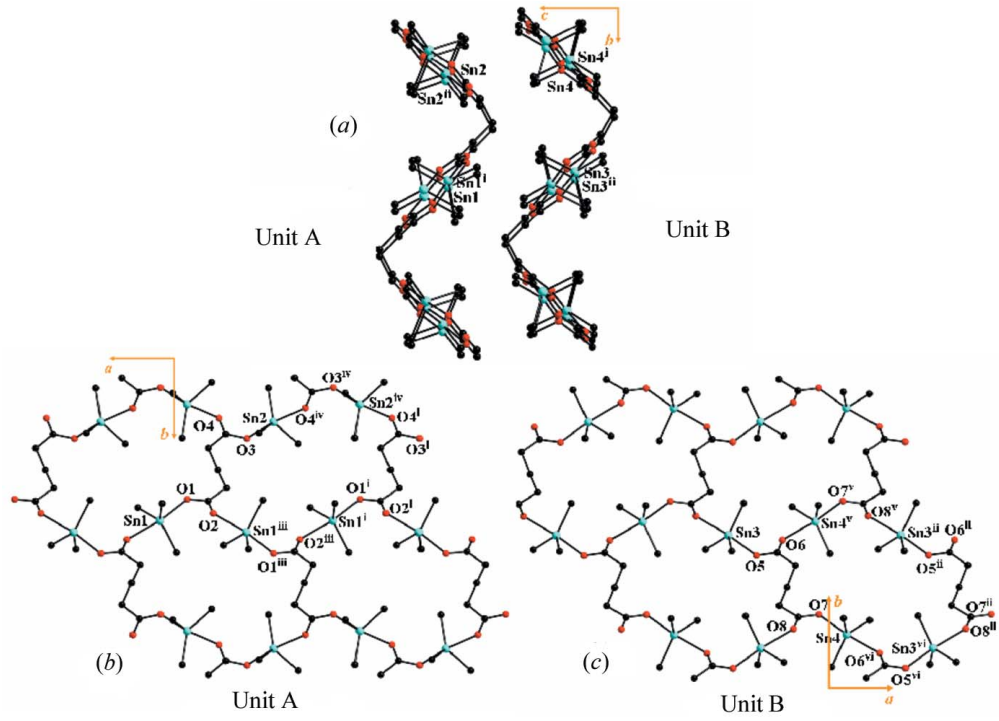

Figure 2
(*a*) A view along the *a* axis of the structure of (I), showing the two units. (*b*) A view along the *b* axis of unit A, showing the two-dimensional sheet incorporating Sn1 and Sn2. (*c*) A view along the *b* axis of unit B, showing the two-dimensional sheet incorporating Sn3 and Sn4. [Symmetry codes: (i) $x-1, y, z$; (ii) $x+1, y, z$; (iii) $x-\frac{1}{2}, \frac{3}{2}-y, 2-z$; (iv) $x-\frac{1}{2}$,1/2 − y, 2 − z; (v)-x,1/2 + y, $\frac{3}{2}-z$; (vi)-x, $y-\frac{1}{2}, \frac{3}{2}-z$.]

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