metal-organic papers

catena-Poly[[trimethyltin(IV)]-μ-3-(trimethylstannylsulfanyl)benzoato-κ²O:O′]

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The title compound, more simply known as 3-mercaptobenzoatobis(trimethyltin), $[Sn_2(CH_3)_6(C_7H_4O_2S)]_n$, is a helical carboxylate-bridged polymer that extends along the b axis of the monoclinic unit cell. The S-bonded Sn atom is four-coordinate in a tetrahedral geometry, whereas the ester-bound Sn atom is five-coordinate in a $trans$-C_3SnO_2 trigonal–bipyramidal geometry.

Comment

Trialkyltin(IV) alkanoates and arylcarboxylates generally exist as carboxylate-bridged chains (Tiekink, 1991, 1994); the conformation of the chain depends on how one molecule is related to the next, but is usually a helix owing to propagation by twofold screw axes. The nature of the organic group of the carboxylic acid has little effect on the repeat distance of the polymer (Ng *et al.*, 1988). Bis(trimethyltin) 3-mercaptobenzoate, (I) (Fig. 1), exists as such a carboxylate-bridged helical polymer; the repeat distance, which is the b axis length, falls within the range found in trialkyltin and triaryltin carboxylates (Ng *et al.*, 1988). The ester-bound Sn atom shows $trans$-C_3SnO_2 trigonal–bipyramidal coordination and the two axial bonds are significantly different, the formally dative Sn—O bond being much longer than the covalent Sn—O bond (Table 1).

(I)
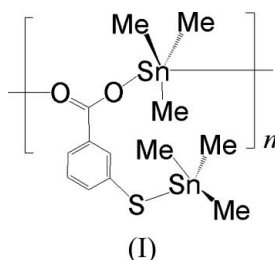
The Sn—S bond distance compares well with the distance found in tetrahedral sulfanyltin compounds such as bis-(triphenyltin) 2-mercaptobenzoate (Ng, Chin *et al.*, 1989) and triphenyltin 2-aminophenylsulfide (Ng, Kumar Das *et al.*, 1989); the sulfide linkage decreases the Lewis acidity of the metal atom so that it is not likely to have a coordination number greater than five.

Experimental

The synthesis of (I) was carried out under a nitrogen atmosphere. 3-Mercaptobenzoic acid (0.15 g, 1 mmol) was added to a benzene solution (20 ml) of sodium ethoxide (0.14 g, 2 mmol). After the acid had dissolved, trimethyltin chloride (0.40 g, 2 mmol) was added and the mixture was heated for 12 h at 313 K. The solution was filtered to remove the sodium chloride precipitate and the solvent was then allowed to evaporate. Colorless crystals were obtained in 80% yield

(m.p. 378 K). Analysis calculated for $C_{13}H_{22}O_2SSn_2$: C 32.54, H 4.62%; found C 32.50, H 4.65%. (**Caution**: trimethyltin chloride is toxic and it should be handled in an efficient fume hood.)

Crystal data

$[Sn_2(CH_3)_6(C_7H_4O_2S)]$	$D_x = 1.766$ Mg m^{-3}
$M_r = 479.75$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 5208
$a = 15.166$ (1) Å	reflections
$b = 9.9706$ (9) Å	$\theta = 2.5–28.3°$
$c = 12.909$ (1) Å	$\mu = 2.88$ mm^{-1}
$\beta = 112.398$ (1)°	$T = 295$ (2) K
$V = 1804.7$ (3) Å^3	Block, colorless
$Z = 4$	$0.39 \times 0.35 \times 0.31$ mm

Data collection

Bruker SMART area-detector diffractometer	4364 independent reflections
	3322 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.025$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$\theta_{max} = 28.3°$
	$h = -17 \rightarrow 19$
$T_{min} = 0.223$, $T_{max} = 0.410$	$k = -13 \rightarrow 13$
11429 measured reflections	$l = -16 \rightarrow 10$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.037P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.026$	$+ 0.7735P]$
$wR(F^2) = 0.077$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.001$
4364 reflections	$\Delta\rho_{max} = 0.68$ e Å^{-3}
169 parameters	$\Delta\rho_{min} = -0.61$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Sn1—C1	2.121 (4)	Sn2—C6	2.114 (5)
Sn1—C2	2.120 (4)	Sn1—O1	2.153 (2)
Sn1—C3	2.113 (4)	Sn1—O2^i	2.465 (2)
Sn2—C4	2.126 (5)	Sn2—S1	2.440 (1)
Sn2—C5	2.142 (5)		
C1—Sn1—C2	114.3 (2)	C3—Sn1—O2^i	83.9 (1)
C1—Sn1—C3	125.0 (2)	O1—Sn1—O2^i	172.8 (1)
C1—Sn1—O1	98.5 (1)	C4—Sn2—C5	113.7 (2)
C1—Sn1—O2^i	87.5 (1)	C4—Sn2—C6	113.8 (2)
C2—Sn1—C3	118.9 (2)	C4—Sn2—S1	104.1 (1)
C2—Sn1—O1	88.5 (1)	C5—Sn2—C6	113.0 (2)
C2—Sn1—O2^i	85.4 (1)	C5—Sn2—S1	108.7 (1)
C3—Sn1—O1	95.8 (1)	C6—Sn2—S1	102.6 (1)

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

H atoms were positioned geometrically [aromatic C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$; methyl C—H = 0.96 Å and $U_{iso}(H) = 1.2U_{eq}(C)$], and were included in the refinement in the riding-model approximation. The methyl groups were rotated to fit the electron density.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve
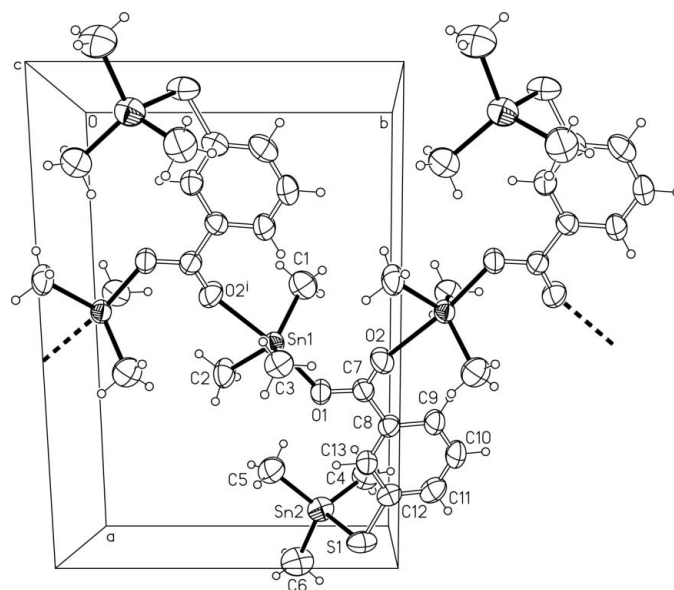

Figure 1
ORTEPII (Johnson, 1976) plot of the chain structure of polymeric (I). Displacement ellipsoids are shown at the 50% probability level and H atoms as spheres of arbitrary radii. [Symmetry code: (i) $1 - x$, $y - \frac{1}{2}$, $\frac{1}{2} - z$.]

structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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