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[(*N*,*N*-Dimethylthiocarbamoylthio)acetato]-trimethyltin

Seik Weng Ng[a] and V. G. Kumar Das[b]

[a]*Institute of Advanced Studies, University of Malaya, 59100 Kuala Lumpur, Malaysia, and* [b]*Department of Chemistry, University of Malaya, 59100 Kuala Lumpur, Malaysia. E-mail: hlnswen@cc.um.edu.my*

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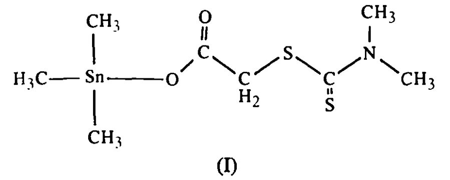
Abstract

Carboxy bridges link the independent molecules of the title compound, *catena*-poly[trimethyltin-μ-(*N*,*N*-dimethylthiocarbamoylthio)acetato-*O*:*O*′], [Sn-($C_5H_8NO_2S_2$)(CH_3)$_3$], in a linear chain along the *x* axis; both five-coordinate Sn atoms (two independent molecules in the asymmetric unit) show *trans*-trigonal bipyramidal coordination.

Comment

[(*N*,*N*-Dimethylthiocarbamoylthio)acetato] trimethyltin and [(*N*,*N*-dimethylthiocarbamoylthio)acetato]triphenyltin are predicted to adopt helical chain structures on the basis of variable-temperature ^{119m}Sn Mössbauer measurements (Ng & Kumar Das, 1991), and the assignment for the triphenyltin ester, which propagates by twofold screw axes, has been been confirmed recently (Ng & Kumar Das, 1995).

[(*N*,*N*-Dimethylthiocarbamoylthio)acetato]trimethyltin, (I), crystallizes as two symmetry-independent molecules linked by carboxylate bridges into a chain, which propagates by translations along *x*. Both Sn atoms show *trans*-C_3SnO_2 trigonal bipyramidal coordination and bond dimensions involving the Sn atoms compare well with those found in other carboxylate-bridged trimethyltin esters (Ng, Chen & Kumar Das, 1988; Tiekink, 1991). Trimethyltin acetate, trimethyltin trifluoroacetate (Chih & Penfold, 1973) and trimethyltin thiophene-2-carboxylate (Sandhu, Verma & Tiekink, 1990) propagate by glides to give rise to zigzag chains. The furan-2-carboxylate (Tiekink, Sandhu & Verma, 1989) and substituted benzoates (Holmes, Day, Chandrasekhar & Holmes, 1986; Smith, Day, Chandrasekhar, Holmes & Holmes, 1986) propagate by twofold screw axes to form stretched helical chains. The (*N*,*N*-dimethylthiocarbamoylthio)acetate only propagates by translations and forms more of a zigzag than a helical chain.



(I)

The C—O bond distances in the carboxylate anion of molecule *A* are differentiated into single- and double-bond distances [C—O 1.265 (4), C=O 1.231 (4) Å]; the anion forms a covalent [2.234 (2) Å] bond to the Sn1*A* and a bridge [2.433 (2) Å] to the Sn1*B* atoms. On the other hand, those in molecule *B* are equal [C—O 1.257 (4), C=O 1.250 (4) Å] and the covalent bonding [2.205 (2) Å] and bridging [2.363 (2) Å] bond distances involving this anion are relatively equal. A similar isobidentate/anisobidentate bonding mode has been documented for the formate anion in the two independent molecules of triphenyltin formate (Molloy, Quill & Nowell, 1987).

Other bond dimensions of the carboxylate anion are similar to those found in the tris(hydroxymethyl)-methylammonium (Ng, 1995) and hydrated dicyclohexylammonium (Ng, 1992) salts.

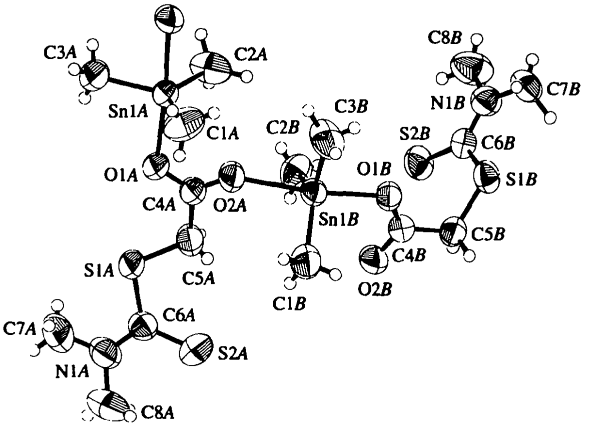


Fig. 1. Atomic labelling scheme for the two independent molecules of the title compound. Displacement ellipsoids are plotted at the 70% probability level.

Experimental

The title compound was synthesized from trimethyltin chloride and sodium (*N*,*N*-dimethylthiocarbamoylthio)acetate (Ng & Kumar Das, 1991), and crystals were grown from its solution in ethanol.

Crystal data

[Sn(C$_5$H$_8$NO$_2$S$_2$)(CH$_3$)$_3$]
$M_r = 342.04$
Triclinic
$P\bar{1}$
$a = 9.761\,(2)$ Å
$b = 12.422\,(2)$ Å
$c = 12.486\,(2)$ Å
$\alpha = 108.27\,(1)°$
$\beta = 101.26\,(2)°$
$\gamma = 100.02\,(2)°$
$V = 1364.3\,(4)$ Å^3
$Z = 4$
$D_x = 1.665$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 14–15°$
$\mu = 2.158$ mm^{-1}
$T = 298$ K
Block
0.40 × 0.40 × 0.25 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
$T_{\rm min} = 0.837$, $T_{\rm max} = 1.000$
8375 measured reflections
7938 independent reflections

6548 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.0313$
$\theta_{\rm max} = 29.97°$
$h = 0 \rightarrow 13$
$k = -17 \rightarrow 17$
$l = -17 \rightarrow 17$
3 standard reflections frequency: 60 min intensity decay: 7.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0385$
$wR(F^2) = 0.0991$
$S = 1.095$
7938 reflections
355 parameters
H atoms were located and refined with U fixed at 0.1 Å^2
$w = 1/[\sigma^2(F_o^2) + (0.0602P)^2 + 0.1270P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = -0.001$
$\Delta\rho_{\rm max} = 1.806$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.659$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Sn1A	1.28582 (2)	0.23808 (2)	0.24316 (2)	0.04002 (7)
S1A	0.9839 (1)	0.52150 (9)	0.1742 (1)	0.0657 (3)
S2A	0.7536 (2)	0.5901 (1)	0.2885 (2)	0.0956 (5)
O1A	1.1347 (2)	0.3446 (2)	0.2024 (2)	0.0526 (6)
O2A	0.9980 (3)	0.2695 (2)	0.2949 (2)	0.0551 (6)
N1A	0.8842 (3)	0.7029 (3)	0.1762 (3)	0.0588 (7)
C1A	1.3384 (6)	0.3335 (5)	0.4231 (4)	0.071 (1)
C2A	1.1313 (5)	0.0765 (4)	0.1495 (5)	0.068 (1)
C3A	1.4149 (5)	0.2890 (5)	0.1411 (5)	0.069 (1)
C4A	1.0242 (3)	0.3344 (3)	0.2406 (3)	0.0483 (7)
C5A	0.9153 (5)	0.4014 (5)	0.2154 (6)	0.083 (2)
C6A	0.8695 (3)	0.6129 (3)	0.2130 (3)	0.0481 (7)
C7A	0.9847 (7)	0.7236 (6)	0.1087 (8)	0.096 (2)
C8A	0.8020 (8)	0.7897 (5)	0.2064 (7)	0.095 (2)
Sn1B	0.79145 (2)	0.16842 (2)	0.33868 (2)	0.04185 (7)
S1B	0.3831 (1)	−0.15048 (7)	0.35545 (7)	0.0491 (2)
S2B	0.3661 (1)	−0.16700 (8)	0.10749 (8)	0.0563 (2)
O1B	0.6138 (2)	0.0559 (2)	0.3695 (2)	0.0438 (5)
O2B	0.4638 (2)	0.1423 (2)	0.2928 (2)	0.0518 (5)
N1B	0.3823 (3)	−0.3391 (2)	0.1895 (2)	0.0518 (6)
C1B	0.7676 (5)	0.3321 (4)	0.4388 (4)	0.066 (1)
C2B	0.7141 (5)	0.0847 (4)	0.1559 (3)	0.0586 (9)
C3B	0.9448 (5)	0.1096 (5)	0.4349 (5)	0.076 (1)
C4B	0.4887 (3)	0.0665 (3)	0.3350 (3)	0.0405 (6)
C5B	0.3636 (4)	−0.0110 (3)	0.3521 (3)	0.0467 (7)
C6B	0.3783 (3)	−0.2280 (3)	0.2087 (3)	0.0418 (6)
C7B	0.3876 (5)	−0.3905 (4)	0.2796 (4)	0.0613 (9)
C8B	0.3799 (7)	−0.4166 (4)	0.0746 (4)	0.079 (1)

Table 2. *Selected geometric parameters (Å, °)*

Sn1A—C1A	2.095 (4)	Sn1B—C1B	2.106 (4)
Sn1A—C2A	2.111 (4)	Sn1B—C2B	2.104 (4)
Sn1A—C3A	2.113 (4)	Sn1B—C3B	2.109 (4)
Sn1A—O1A	2.234 (2)	Sn1B—O1B	2.205 (2)
Sn1A—O2B$^{\rm i}$	2.363 (2)	S1B—C5B	1.788 (3)
S1A—C5A	1.791 (4)	S1B—C6B	1.770 (3)
S1A—C6A	1.755 (3)	S2B—C6B	1.661 (3)
S2A—C6A	1.649 (4)	O1B—C4B	1.257 (4)
O1A—C4A	1.265 (4)	O2B—C4B	1.250 (4)
O2A—C4A	1.231 (4)	O2B—Sn1A$^{\rm ii}$	2.363 (2)
O2A—Sn1B	2.433 (2)	N1B—C6B	1.333 (4)
N1A—C6A	1.332 (4)	N1B—C7B	1.454 (5)
N1A—C7A	1.453 (7)	N1B—C8B	1.449 (5)
N1A—C8A	1.453 (6)	C4B—C5B	1.512 (4)
C4A—C5A	1.503 (5)		
C1A—Sn1A—C2A	127.0 (2)	C1B—Sn1B—C2B	126.3 (2)
C1A—Sn1A—C3A	119.5 (2)	C1B—Sn1B—C3B	112.3 (2)
C1A—Sn1A—O1A	92.3 (2)	C1B—Sn1B—O1B	98.2 (1)
C1A—Sn1A—O2B$^{\rm i}$	85.3 (2)	C1B—Sn1B—O2A	89.1 (1)
C2A—Sn1A—C3A	112.8 (2)	C2B—Sn1B—C3B	120.1 (2)
C2A—Sn1A—O1A	94.0 (1)	C2B—Sn1B—O1B	92.5 (1)
C2A—Sn1A—O2B$^{\rm i}$	91.5 (1)	C2B—Sn1B—O2A	84.5 (1)
C3A—Sn1A—O1A	91.4 (1)	C3B—Sn1B—O1B	90.7 (2)
C3A—Sn1A—O2B$^{\rm i}$	85.4 (1)	C3B—Sn1B—O2A	84.8 (2)
O1A—Sn1A—O2B$^{\rm i}$	174.44 (8)	O1B—Sn1B—O2A	172.49 (8)
C5A—S1A—C6A	101.6 (2)	C5B—S1B—C6B	102.6 (2)
C4A—O1A—Sn1A	115.4 (2)	C4B—O1B—Sn1B	116.8 (2)
C4A—O2A—Sn1B	138.9 (2)	C4B—O2B—Sn1A$^{\rm ii}$	143.5 (2)
C6A—N1A—C7A	123.3 (3)	C6B—N1B—C7B	123.3 (3)
C6A—N1A—C8A	120.6 (4)	C6B—N1B—C8B	120.8 (3)
C7A—N1A—C8A	116.0 (4)	C7B—N1B—C8B	115.9 (3)
O1A—C4A—O2A	123.4 (3)	O1B—C4B—O2B	123.0 (3)
O1A—C4A—C5A	119.0 (3)	O1B—C4B—C5B	118.5 (3)
O2A—C4A—C5A	117.6 (3)	O2B—C4B—C5B	118.4 (3)
C4A—C5A—S1A	113.8 (3)	C4B—C5B—S1B	117.3 (2)
N1A—C6A—S1A	114.1 (3)	N1B—C6B—S1B	112.6 (2)
N1A—C6A—S2A	122.6 (3)	N1B—C6B—S2B	124.9 (2)
S1A—C6A—S2A	123.3 (2)	S1B—C6B—S2B	122.4 (2)

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

Data collection: *CAD*-4 *VAX*/*PC* (Enraf–Nonius, 1988). Cell refinement: *CAD*-4 *VAX*/*PC*. Data reduction: *Xtal*3.0 (Hall & Stewart, 1990). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai & Pritzkow, 1994). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1073). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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[3-(Diethylphosphono)propionato]triphenyltin

Seik Weng Ng[a] and V. G. Kumar Das[b]

[a]*Institute of Advanced Studies, University of Malaya, 59100 Kuala Lumpur, Malaysia, and* [b]*Department of Chemistry, University of Malaya, 59100 Kuala Lumpur, Malaysia. E-mail: h1nswen@cc.um.edu.my*

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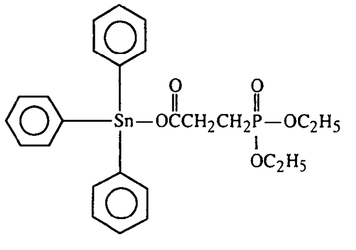
Abstract

In the title compound, $[Sn(C_7H_{14}O_5P)(C_6H_5)_3]$, the planar triphenyltin cations are axially bridged through the carboxy and phosphoryl O atoms of the 3-(diethylphosphono)propionate anions into zigzag chains that run parallel to the *c* axis {*i.e. catena*-poly[triphenyltin-μ-3-(diethylphosphono)propionato-O:$O^{P=O}$]}.

Comment

Triphenyltin alkanoates generally adopt carboxylate-bridged structures in which the triphenyltin cations are linked axially by the carboxylate anions into linear chains (Ng, Chen & Kumar Das, 1988; Tiekink, 1991, 1994). A second donor O atom in the carboxylate anion allows the possibility of bridging through this site, a feature that has been documented for (diethylphosphonoacetato)triphenyltin [Sn—O = 2.129 (3), Sn←O = 2.420 (3) Å], which propagates by translations along the *b* axis (Ng & Kumar Das, 1994). The introduction of another methylene linkage in this ester results in no significant differences in the coordination geometry of the Sn atom in the title compound, (I) [Sn—O = 2.116 (2), Sn←O = 2.397 (3) Å]. However, the 3-(diethylphosphono)propionate propagates the chain by a glide along the *c* axis, so that the chain has a zigzag conformation.



(I)

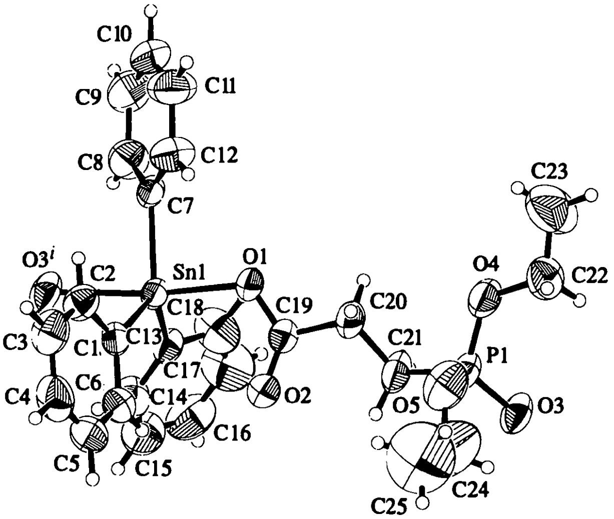


Fig. 1. Atomic labelling scheme for the title compound. *ZORTEP* (Zsolnai & Pritzkow, 1994) displacement ellipsoids are plotted at the 50% probability level.

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