Data collection: *CAD*-4-*VAX*/*PC* (Enraf–Nonius, 1988). Cell refinement: *CAD*-4-*VAX*/*PC*. Data reduction: *NRCVAX* (Gabe, Le Page, Charland, Lee & White (1989). Program(s) used to solve structure: *SHELXL*93 (Sheldrick, 1993). Program(s) used to refine structure: *SHELXL*93. Molecular graphics: *ZORTEP* (Zsolnai & Pritzkow, 1994). Software used to prepare material for publication: *SHELXL*93.

We thank the University of Malaya (PJP 65/95) for supporting this work.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1077). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Diisopropylammonium (Mercaptoacetato-*S*,*O*)triphenylstannate

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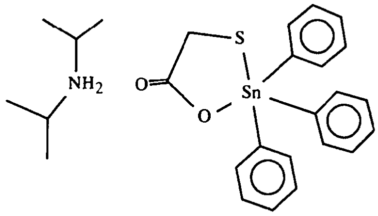
Abstract

In the title compound, $[NH_2(C_3H_7)_2][Sn(C_2H_2O_2S)(C_6H_5)_3]$, the mercaptoacetate dianion chelates to the Sn atom [Sn—S = 2.432 (1), Sn←O = 2.608 (3) Å, S—Sn—O = 74.2 (1)°], which displays *cis*-C_3SnOS trigonal bipyramidal coordination. Two stannate ions are linked together through two ammonium cations [N···O = 2.753 (5), 2.886 (5) Å] to give rise to a dimeric ion pair.

Comment

Triphenyltin mercaptides generally display tetrahedral coordination at the Sn atom as the Sn—S bond reduces the Lewis acidity of the triphenyltin cation, but in dicyclohexylammonium (2-mercaptobenzoato)triphenylstannate the carboxy entity engages in intramolecular coordination [Sn—O = 2.704 (3) Å] to tin. Hydrogen bonds [N···O = 2.803 (4), 2.880 (4) Å] from the cation link two ion pairs into a centrosymmetric dimer (Ng & Kumar Das, 1993). For the (mercaptoacetato)triphenylstannate analogue (Ng, Chin, Chen, Kumar Das & Mak, 1989), Mössbauer measurements also implied a *cis*-C_3SnOS trigonal bipyramidal geometry at Sn, but the insolubility of the compound in common organic solvents precluded its crystallographic verification.

The Sn atom in diisopropylammonium triphenyl(mercaptoacetato-*O*,*S*)stannate, (I), is five-coordinate in a *cis*-trigonal bipyramidal polyhedron whose apical positions are occupied by the C13 and O1 atoms [Sn—O = 2.608 (3) Å]. The Sn1—S1—C19—C20—O1 ring is not planar [Σ = 525 (1)°], and the S1—C19—C20 angle [115.1 (4)°] has been widened from the tetrahedral angle of 109.5°. Of the three Sn—C bonds, the axial bond [2.177 (5) Å] is statistically longer than the two equatorial bonds [2.144 (4), 2.146 (4) Å].



(I)

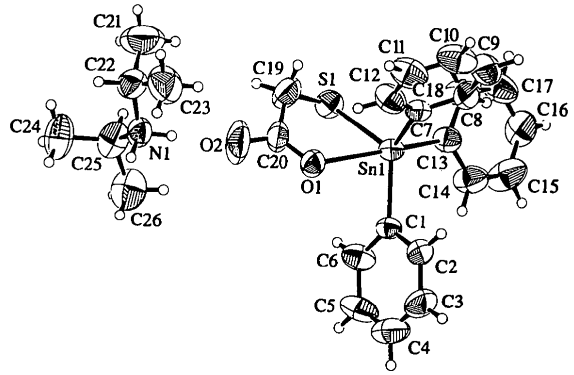


Fig. 1. Atomic labelling scheme for the title compound; displacement ellipsoids are plotted at the 50% probability level (*ZORTEP*; Zsolnai & Pritzkow, 1994).

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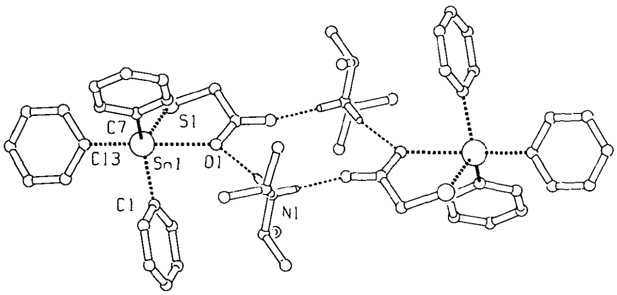

Fig. 2. *PLUTON* (Spek, 1994) plot of the dimeric ion pair showing the hydrogen-bonding interactions.

Experimental

Triphenyltin hydroxide was added to an ethanol solution containing equimolar amounts of diisopropylamine and mercaptoacetic acid. The mixture was briefly heated; slow cooling of the filtered solution gave large crystals of the title complex.

Crystal data

$(C_6H_{16}N)[Sn(C_2H_2O_2S)(C_6H_5)_3]$
$M_r = 542.28$
Monoclinic
$P2_1/n$
$a = 9.482\,(2)$ Å
$b = 17.146\,(3)$ Å
$c = 16.931\,(4)$ Å
$\beta = 98.72\,(2)°$
$V = 2721\,(1)$ Å^3
$Z = 4$
$D_x = 1.324$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10.0$–$12.5°$
$\mu = 1.036$ mm^{-1}
$T = 298$ K
Cube
$0.40 \times 0.40 \times 0.40$ mm
Colourless

Data collection

Siemens *P*4 diffractometer
ω-2θ scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
$T_{\min} = 0.710$, $T_{\max} = 0.841$
4162 measured reflections
4014 independent reflections

3081 observed reflections $[I > 2\sigma(I)]$
$R_{\rm int} = 0.0213$
$\theta_{\max} = 23.5°$
$h = -10 \rightarrow 10$
$k = 0 \rightarrow 19$
$l = 0 \rightarrow 18$
3 standard reflections frequency: 120 min intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0351$
$wR(F^2) = 0.0834$
$S = 1.020$
4014 reflections
280 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0380P)^2 + 0.7588P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.390$ e Å^{-3}
$\Delta\rho_{\min} = -0.258$ 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*	$U_{\rm eq}$
Sn1	0.07996 (3)	0.17377 (2)	0.92420 (2)	0.0450 (1)
S1	0.1816 (1)	0.25615 (8)	0.83030 (8)	0.0611 (3)
O1	0.0535 (3)	0.3166 (2)	0.9729 (2)	0.0601 (9)
O2	0.1071 (5)	0.4374 (2)	0.9524 (3)	0.127 (2)
N1	0.1298 (4)	0.5966 (2)	0.9375 (2)	0.063 (1)
C1	−0.1430 (4)	0.1794 (3)	0.9332 (2)	0.045 (1)
C2	−0.1937 (5)	0.1422 (3)	0.9954 (3)	0.064 (1)
C3	−0.3383 (7)	0.1421 (4)	1.0015 (4)	0.087 (2)
C4	−0.4330 (6)	0.1787 (4)	0.9458 (4)	0.090 (2)
C5	−0.3876 (6)	0.2157 (4)	0.8831 (4)	0.082 (2)
C6	−0.2422 (5)	0.2164 (3)	0.8768 (3)	0.067 (1)
C7	0.2218 (4)	0.1556 (3)	1.0340 (3)	0.047 (1)
C8	0.2706 (5)	0.0806 (3)	1.0533 (3)	0.063 (1)
C9	0.3635 (6)	0.0654 (4)	1.1239 (4)	0.082 (2)
C10	0.4074 (6)	0.1252 (5)	1.1756 (3)	0.086 (2)
C11	0.3621 (6)	0.1996 (4)	1.1570 (4)	0.085 (2)
C12	0.2702 (5)	0.2142 (3)	1.0870 (3)	0.068 (1)
C13	0.0902 (5)	0.0666 (3)	0.8564 (3)	0.053 (1)
C14	−0.0303 (6)	0.0220 (3)	0.8334 (3)	0.081 (2)
C15	−0.0283 (7)	−0.0474 (4)	0.7923 (4)	0.103 (2)
C16	0.0982 (7)	−0.0744 (3)	0.7735 (4)	0.086 (2)
C17	0.2193 (6)	−0.0326 (4)	0.7949 (3)	0.089 (2)
C18	0.2156 (5)	0.0370 (3)	0.8359 (3)	0.079 (2)
C19	0.2298 (6)	0.3420 (3)	0.8905 (3)	0.079 (2)
C20	0.1202 (5)	0.3679 (3)	0.9419 (3)	0.061 (1)
C21	0.3889 (7)	0.5908 (4)	0.9268 (5)	0.133 (3)
C22	0.2786 (6)	0.6185 (3)	0.9771 (4)	0.080 (2)
C23	0.3003 (7)	0.5835 (4)	1.0599 (4)	0.109 (2)
C24	0.0900 (8)	0.7081 (3)	0.8440 (4)	0.102 (2)
C25	0.0840 (6)	0.6203 (3)	0.8523 (3)	0.080 (2)
C26	−0.0650 (7)	0.5877 (4)	0.8259 (4)	0.106 (2)

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

Sn1—C1	2.144 (4)	Sn1—O1	2.608 (3)
Sn1—C7	2.146 (4)	N1—O1$^{\rm i}$	2.886 (5)
Sn1—C13	2.177 (5)	N1—O2	2.753 (5)
Sn1—S1	2.432 (1)		
C1—Sn1—C7	116.1 (2)	C7—Sn1—S1	113.1 (1)
C1—Sn1—C13	101.5 (2)	C7—Sn1—O1	86.6 (1)
C1—Sn1—S1	120.8 (1)	C13—Sn1—S1	95.2 (1)
C1—Sn1—O1	78.0 (1)	C13—Sn1—O1	166.8 (1)
C7—Sn1—C13	105.2 (2)	S1—Sn1—O1	74.2 (1)

Symmetry code: (i) $-x, 1 - y, 2 - z$.

Each H atom was refined with $U_{\rm eq}$ equal to $1.5U_{\rm eq}$ of the parent atom.

Data collection: *XSCANS* (Fait, 1991). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *PLUTON* (Spek, 1994), *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: KH1078). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

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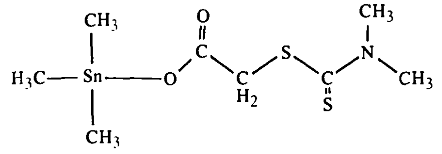
Abstract

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.

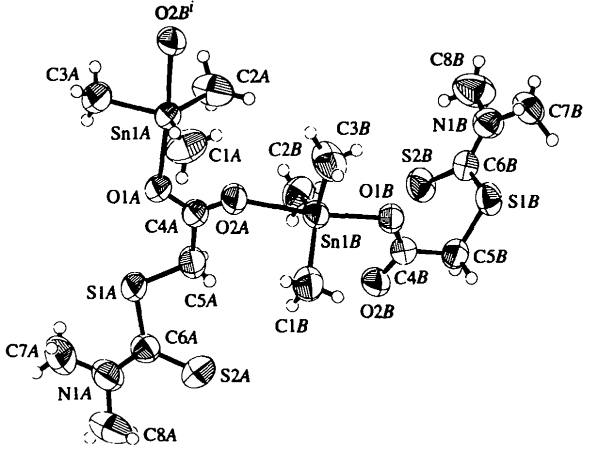


Fig. 1. Atomic labelling scheme for the two independent molecules of the title compound. Displacement ellipsoids are plotted at the 70% probability level.