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Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1237). Services for accessing these data are described at the back of the journal.

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Diisopropylammonium oxalatotriphenylstannate

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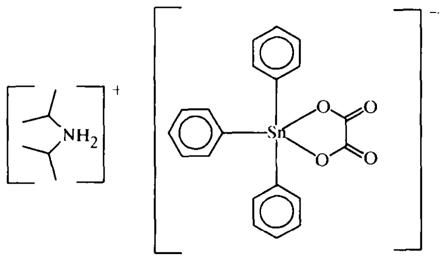
Abstract

The coordination polyhedron of the five-coordinate Sn atom in the title compound, diisopropylammonium (oxalato-O^1,O^2)triphenylstannate, $[NH_2(C_3H_7)_2][Sn(C_6H_5)_3(C_2O_4)]$, is a *cis*-$C_3SnO_2$ trigonal bipyramid [Sn—O_{eq} = 2.115 (1) and Sn—O_{ax} = 2.260 (1) Å] that is approximately 50% displaced towards the square pyramid along the Berry pseudo-rotation pathway. Two cations and two anions participate in a hydrogen-bonded cluster [N···O 2.808 (3)–3.044 (3) Å]

Comment

With few exceptions, five-coordinate triorganotin compounds adopt a trigonal-bipyramidal configuration at the Sn atom, and examples of the *trans*-C_3SnXY form vastly outnumber those of the *cis*-C_3SnXY form. The latter arrangement can be effected by the use of a bidentate monoanionic group; the use of a bidentate dianionic group introduces the additional influence of a counterion, whose effect on the structure of the onium triorganostannate may be difficult to predict *a priori*. The oxalato group chelates to the triphenyltin cation in the dicyclohexylammonium derivative (Ng & Kumar Das, 1995), but it merely links adjacent triphenyltin girdles in a linear chain in the trimethylsulfonium salt (Ng *et al.*, 1994). On the other hand, the ligand exhibits both binding abilities in the 1/1 complex of tetramethylammonium oxalatotriphenylstannate with bis(triphenyltin) oxalate (Ng & Kumar Das, 1993).

The geometry of the Sn atom in the title compound, (I), is a *cis*-trigonal bipyramid and bond dimensions involving the Sn atom are similar to those found in the bis(dicyclohexylammonium) salt, which crystallizes in a triclinic as well as in a monoclinic modification (Ng & Kumar Das, 1995). The geometry is displaced by 55% along the tbp–sq (trigonal bipyramid–square pyramid) Berry pseudo-rotation coordinate (Spek, 1990), which exceeds the average value of 30% found for the two independent ion pairs in the triclinic bis(dicyclohexylammonium) salt. The hydrogen-bonding interactions [N···O = 2.808 (3) and 2.864 (3) Å] are similar to those found in bis(diisopropylammonium) bis(mercaptoacetatotriphenylstannate) [2.753 (5) and 2.886 (5) Å], whose Sn atom is *O*,*S*-chelated by the anionic group (Ng *et al.*, 1996).



(I)

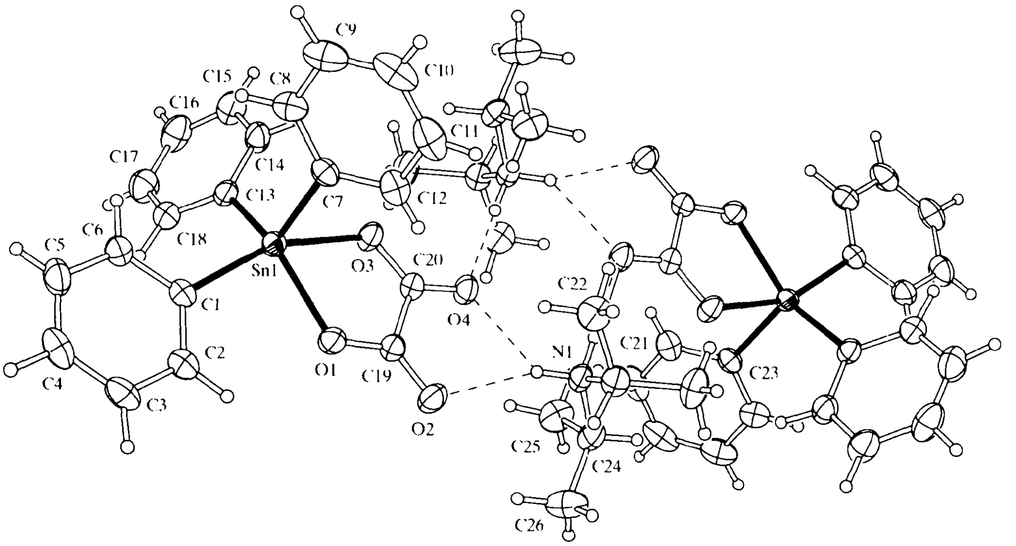


Fig. 1. *ORTEP*II (Johnson, 1976) plot of the title compound at the 30% probability level. H atoms are drawn as spheres of arbitrary radii.

Experimental

Compound (I) was synthesized by the reaction of diisopropylamine, triphenyltin hydroxide and oxalic acid dihydrate (1:1:1 molar ratio). The reagents were heated in a small volume of ethanol and compound (I) separated on cooling the filtered solution. Solid-state ^{119}Sn NMR (relative to Me_4Sn): two sites centered at −199 and −213 p.p.m.

Crystal data

$(C_6H_{16}N)[Sn(C_6H_5)_3(C_2O_4)]$
$M_r = 540.23$
Triclinic
$P\bar{1}$
$a = 10.3121\ (4)$ Å
$b = 11.3409\ (7)$ Å
$c = 12.7787\ (8)$ Å
$\alpha = 113.224\ (5)°$
$\beta = 101.056\ (4)°$
$\gamma = 102.401\ (4)°$
$V = 1276.1\ (1)$ Å^3
$Z = 2$
$D_x = 1.406$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 12.0–13.0°$
$\mu = 1.031$ mm^{-1}
$T = 298\ (2)$ K
Irregular block
0.35 × 0.30 × 0.25 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.668$, $T_{\max} = 0.773$
7790 measured reflections
7406 independent reflections

6320 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.009$
$\theta_{\max} = 29.97°$
$h = 0 \rightarrow 14$
$k = -15 \rightarrow 15$
$l = -17 \rightarrow 17$
3 standard reflections frequency: 60 min intensity decay: 4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.066$
$S = 0.999$
7406 reflections
413 parameters
H atoms were located and refined

$w = 1/[\sigma^2(F_o^2) + (0.0386P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.312$ e Å^{-3}
$\Delta\rho_{\min} = -0.320$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Sn1—C1	2.159 (2)	Sn1—O1	2.115 (1)
Sn1—C7	2.134 (2)	Sn1—O3	2.260 (1)
Sn1—C13	2.146 (2)		
C1—Sn1—C7	106.9 (1)	C7—Sn1—O1	103.1 (1)
C1—Sn1—C13	103.9 (1)	C7—Sn1—O3	88.2 (1)
C1—Sn1—O1	88.4 (1)	C13—Sn1—O1	134.7 (1)
C1—Sn1—O3	158.7 (1)	C13—Sn1—O3	82.5 (1)
C7—Sn1—C13	114.1 (1)	O1—Sn1—O3	73.3 (1)

Data collection: *CAD*-4/*PC* (Kretschmar, 1994). Cell refinement: *CAD*-4 *VAX*/*PC* (Enraf–Nonius, 1988). Data reduction: *XCAD*4 (Harms, 1997). Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*b*). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1232). Services for accessing these data are described at the back of the journal.

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Bis[(*N*,*N*-3-oxapentamethylenethiocarbamoylthioacetato)triphenyltin] hydrate and bis(dicyclohexylammonium) bis(3-oxapentamethylenethiocarbamoylthioacetate)†

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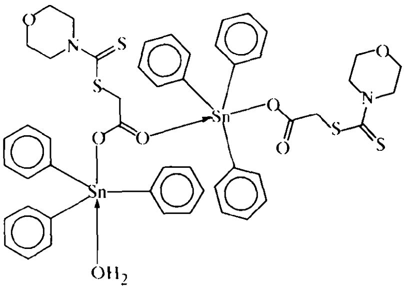
(*Received 31 July 1998; accepted 9 October 1998*)

Abstract

In the two independent dinuclear molecules of bis[(*N*,*N*-3-oxapentamethylenethiocarbamoylthioacetato)triphenyltin] hydrate, $[Sn_2(C_6H_5)_6(C_7H_{10}NO_3S_2)_2(H_2O)]$, the aqua(*N*,*N*-3-oxapentamethylenethiocarbamoylthioacetato)triphenyltin moiety uses its carbonyl O atom to connect to the Sn atom of the (*N*,*N*-3-oxapentamethylenethiocarbamoylthioacetato)triphenyltin moiety, to furnish *trans*-C_3SnO_2 trigonal bipyramidal geometries for the Sn atoms in the dinuclear compound. Hydrogen bonds link the coordinated water of one symmetry-independent molecule to the carbonyl and heterocyclic O atoms of the other independent molecule [O···O = 2.66 (4) and 2.75 (2), and 2.769 (8) and 2.667 (7) Å, respectively] in a layer structure. The orthorhombic unit cell of bis(dicyclohexylammonium) bis(3-oxapentamethylenethiocarbamoylthioacetate), $2C_{12}H_{24}N^+\cdot 2C_7H_{10}NO_3S_2^-$, contains four centrosymmetric clusters of two cations and two anions. The C—O bonds in the carboxyl $-CO_2$ fragment of the anion are delocalized. The ammonium N atom is hydrogen bonded to two carboxyl O atoms belonging to different anions [N···O = 2.732 (4) and 2.738 (4) Å] to form an eight-membered O—C—O···N···O—C—O···N··· ring.

Comment

Triphenyltin thiocarbamoylthioacetates, $[(C_6H_5)_3SnO_2CCH_2SC(S)NR_2]$, have been assigned carboxylate-bridged structures on the basis of spectroscopic measurements (Ng & Kumar Das, 1991). The assignments have been corroborated by crystal structure analysis for the NR_2 = $N(CH_3)_2$ (Ng & Kumar Das, 1995*a*), $N(C_2H_5)_2$ and $N(CH_3)(C_6H_5)$ (Lo *et al.*, 1999) derivatives. The NR_2 = $NO(CH_2CH_2)_2$ derivative, (I), crystallizes with half a water molecule, and there are two independent dinuclear molecules in the unit cell. Each consists of a water-coordinated aqua(*N*,*N*-3-oxapentamethylenethiocarbamoylthioacetato)triphenyltin moiety



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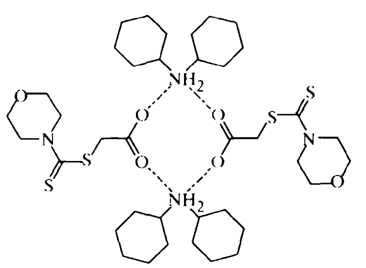

(II)

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† Alternative names: aqua-1κO-μ-[(4-morpholinecarbothioylthio)acetato]-1:2$\kappa^2 O$:O'-[(4-morpholinecarbothioylthio)acetato]-2κO-hexaphenyl-1$\kappa^3 C$,2$\kappa^3 C$-ditin monohydrate and bis(dicyclohexylammonium) bis[(4-morpholinecarbothioylthio)acetate].

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