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Structural studies on triorganostannates derived from dicarboxylic acids. Crystal structure of tetramethylammonium oxalatotriphenylstannate · bis(triphenyltin) oxalate

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Abstract

Triphenyltin chloride reacts with equimolar amounts of tetramethylammonium chloride and of silver oxalate to yield tetramethylammonium bisoxalatotris(triphenylstannate). A crystal structural study has shown the trinuclear stannate to be composed of helical $[\text{Ph}_3\text{SnOx} \cdot (\text{Ph}_3\text{Sn})_2\text{ox}]_n^-$ chains that run parallel to the *b*-axis; the cations fill up the space between the chains. The structure is an unusual example of a triorganotin compound having within the same molecule five-coordinate tin sites in distinct *cis*- and *trans*-trigonal bipyramidal geometries.

1. Introduction

Triorganostannates derived from dicarboxylic acids have been prepared by condensing triorganotin oxides or hydroxides with dicyclohexylammonium hydrogen dicarboxylates. Diffraction studies have shown that their structures are usually more complicated than initially assumed for a 1/1 tin-acid stoichiometry, however. Bis(tributyltin) oxide condenses with two equivalents of dicyclohexylammonium hydrogen oxalate in toluene to give a compound formulated as $[\text{Cyh}_2\text{NH}_2]^+ [\text{Bu}_3\text{SnOx}]^-$, but in ethanol the reaction yields $[\text{Cyh}_2\text{NH}_2]_2^+ [\text{Bu}_3\text{Sn})_2\text{ox}] \cdot [\text{EtOH} \cdot \text{Bu}_3\text{SnOx}]_2^-$. In the crystal structure of the latter the $[\text{Cyh}_2\text{NH}_2]^+$ cations form hydrogen bonds to the dianionic chain, which is composed of two $[\text{EtOH} \cdot \text{Bu}_3\text{SnOx}]^-$ anions coordinated to a $[(\text{Bu}_3\text{Sn})_2\text{ox}]$ molecule [1]. The analogous reaction with dicyclohexylammonium hydrogen malonate in ethanol affords $[\text{Cyh}_2\text{NH}_2]_2^+ [(\text{Bu}_3\text{Sn})_2\text{mal}] \cdot [\text{Bu}_3\text{Snmal}]_2^-$, which contains no ethanol. The dianion in the asymmetric unit in this stannate similarly consists of two $[\text{Bu}_3\text{Snmal}]^-$ anions linked to a $[(\text{Bu}_3\text{Sn})_2\text{mal}]$ molecule; however, a 3-*d* carboxylate-bridged network is formed because both $[\text{Bu}_3\text{Snmal}]^-$ anions are

additionally linked to other $[(\text{Bu}_3\text{Sn})_2\text{mal}]$ molecules [2]. The geometry of the tin sites in the two ammonium stannates is unambiguously *trans*-trigonal bipyramidal. A succinato derivative has been prepared by condensing $[(\text{Bu}_3\text{Sn})_2\text{O}]$ with $[\text{Cyh}_2\text{NH}_2] [\text{Hsuc}]$ in ethanol, but its X-ray structure has not yet been determined.

The simple stannate $[\text{Cyh}_2\text{NH}_2]^+ [\text{Ph}_3\text{SnOx}]^-$ has been prepared by condensing triphenyltin hydroxide with dicyclohexylammonium hydrogen oxalate in ethanol [1]; the geometry of the tin atom in the compound was judged to be *cis*-trigonal bipyramidal on the basis of its Mössbauer spectral data ($IS = 1.02$, $QS = 2.15 \text{ mms}^{-1}$ [3]). The crystal structure of the $[\text{Cyh}_2\text{NH}_2]^+ [\text{Ph}_3\text{Snsuc}]^-$ analogue shows non-chelating succinato groups connecting planar Ph_3Sn cations into chains [4]. The chelating mode of the dicarboxylate dianion has been observed in the diorgano- $[\text{Cyh}_2\text{NH}_2]_2^+ [\text{R}_2\text{SnOx}_2]^{2-}$ [5,6] and monoorgano- $[\text{Cyh}_2\text{NH}_2]_2^+ [\text{RSnOx}_2(\text{O}_2\text{CPh})]^{2-}$ [7] stannates.

In the above series of ammonium stannates the $[\text{Cyh}_2\text{NH}_2]^+$ ion was used as the counter-ion to balance the charges, but the nature of the cation does not appear to be particularly crucial for the crystal structure. To test this point as well as to secure crystallographic documentation for the *cis*-trigonal bipyramidal configuration for $[\text{Ph}_3\text{SnOx}]^-$, we undertook the synthesis of $[\text{Me}_4\text{N}]^+ [\text{Ph}_3\text{SnOx}]^-$ by reaction of equimo-

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TABLE 1. Positional parameters and equivalent isotropic temperature factors^d

Atom	x	y	z	B_{eq} (Å ²)
Sn1	0.26228(6)	0.14934(2)	0.05657(3)	4.35(1)
Sn2	0.19618(6)	0.35221(2)	0.16076(2)	3.92(1)
Sn3	0.39648(6)	0.59699(2)	0.22095(3)	3.84(1)
O1	0.2302(6)	0.2295(2)	0.0972(2)	4.7(1)
O2	0.1593(6)	0.2678(2)	0.1785(2)	4.6(1)
O3	0.1726(6)	0.1416(2)	0.1393(2)	4.7(1)
O4	0.0891(6)	0.1752(2)	0.2202(2)	4.4(1)
O5	0.2142(6)	0.4333(2)	0.1500(3)	5.1(1)
O6	0.3565(7)	0.4409(2)	0.0772(3)	6.2(2)
O7	0.3827(6)	0.5286(2)	0.1699(3)	4.5(1)
O8	0.2063(7)	0.5408(2)	0.0987(3)	6.5(2)
N	0.7654(9)	0.4843(3)	0.0937(4)	6.4(2)
C1	0.2677(9)	0.0685(3)	0.0565(4)	4.8(2)
C2	0.390(1)	0.0437(4)	0.0547(5)	6.8(3)
C3	0.393(1)	-0.0093(4)	0.0567(6)	8.4(3)
C4	0.272(1)	-0.0361(4)	0.0596(5)	7.3(3)
C5	0.150(1)	-0.0115(4)	0.0630(5)	7.3(3)
C6	0.148(1)	0.0417(4)	0.0614(5)	6.4(3)
C7	0.4704(9)	0.1714(3)	0.0585(4)	5.4(2)
C8	0.552(1)	0.1815(5)	0.1134(6)	8.4(3)
C9	0.697(1)	0.1927(5)	0.1123(8)	10.7(4)
C10	0.749(1)	0.1931(5)	0.0553(8)	11.8(5)
C11	0.670(1)	0.1826(6)	0.0038(7)	12.1(4)
C12	0.527(1)	0.1726(5)	0.0040(6)	9.5(3)
C13	0.1212(9)	0.1704(3)	-0.0200(4)	4.8(2)
C14	0.064(1)	0.1323(4)	-0.0574(5)	7.3(3)
C15	-0.030(1)	0.1434(5)	-0.1090(5)	8.8(3)
C16	-0.062(1)	0.1925(5)	-0.1248(5)	8.1(3)
C17	-0.006(1)	0.2314(5)	-0.0878(5)	7.8(3)
C18	0.087(1)	0.2200(4)	-0.0349(4)	6.2(3)
C19	0.0959(8)	0.3654(3)	0.2412(3)	4.0(2)
C20	0.059(1)	0.4137(4)	0.2559(4)	6.5(3)
C21	-0.013(1)	0.4223(4)	0.3070(5)	8.1(3)
C22	-0.044(1)	0.3842(4)	0.3441(5)	7.5(3)
C23	-0.006(1)	0.3365(4)	0.3303(4)	7.0(3)
C24	0.063(1)	0.3270(4)	0.2784(4)	5.6(2)
C25	0.0673(8)	0.3452(3)	0.0770(4)	4.1(2)
C26	-0.058(1)	0.3206(4)	0.0763(5)	6.3(3)
C27	-0.153(1)	0.3199(4)	0.0230(6)	7.4(3)
C28	-0.120(1)	0.3428(4)	-0.0300(5)	7.5(3)
C29	0.003(1)	0.3668(4)	-0.0309(5)	7.4(3)
C30	0.100(1)	0.3681(4)	0.0228(4)	6.0(2)
C31	0.4103(9)	0.3414(3)	0.1762(4)	5.1(2)
C32	0.481(1)	0.3156(5)	0.1376(7)	9.9(4)
C33	0.626(1)	0.3110(6)	0.1509(8)	12.5(5)
C34	0.692(1)	0.3314(6)	0.2041(7)	10.7(4)
C35	0.622(1)	0.3600(7)	0.2381(6)	12.1(5)
C36	0.480(1)	0.3674(6)	0.2244(6)	9.3(4)
C37	0.5325(8)	0.5596(3)	0.2901(4)	4.3(2)
C38	0.6140(9)	0.5871(4)	0.3349(4)	5.7(2)
C39	0.701(1)	0.5612(4)	0.3803(5)	7.0(3)
C40	0.707(1)	0.5096(4)	0.3814(5)	7.4(3)
C41	0.628(1)	0.4825(4)	0.3360(5)	7.3(3)
C42	0.539(1)	0.5072(4)	0.2905(5)	6.2(3)
C43	0.4860(8)	0.6401(3)	0.1532(4)	4.2(2)
C44	0.469(1)	0.6246(4)	0.0921(4)	6.0(2)
C45	0.532(1)	0.6509(4)	0.0468(5)	7.4(3)
C46	0.617(1)	0.6911(4)	0.0628(5)	7.3(3)
C47	0.638(1)	0.7069(4)	0.1245(5)	7.3(3)

TABLE 1 (continued)

Atom	x	y	z	B_{eq} (Å ²)
C48	0.572(1)	0.6811(4)	0.1703(5)	6.2(2)
C49	0.1830(8)	0.6024(3)	0.2293(4)	4.4(2)
C50	0.108(1)	0.6426(4)	0.2027(5)	6.6(3)
C51	-0.033(1)	0.6467(5)	0.2073(5)	9.0(4)
C52	-0.099(1)	0.6102(5)	0.2388(5)	8.2(3)
C53	-0.023(1)	0.5713(5)	0.2666(6)	8.3(3)
C54	0.117(1)	0.5560(4)	0.2621(5)	6.8(3)
C55	0.1805(8)	0.2301(3)	0.1469(3)	3.6(2)
C56	0.1423(8)	0.1788(3)	0.1722(4)	3.9(2)
C57	0.2897(8)	0.4576(3)	0.1157(4)	4.2(2)
C58	0.2892(8)	0.5146(3)	0.1281(3)	4.0(2)
C59	0.698(2)	0.4459(8)	0.056(1)	19.5(7)
C60	0.903(1)	0.4900(8)	0.080(1)	17.7(8)
C61	0.750(3)	0.471(1)	0.1546(8)	19.6(9)
C62	0.689(2)	0.5295(7)	0.080(1)	19.3(8)

^a $B_{eq} = 4/3 (a^2 B_{1,1} + b^2 B_{2,2} + c^2 B_{3,3} + ab(\cos \gamma) B_{1,2} + ac(\cos \beta) B_{1,3} + bc(\cos \alpha) B_{2,3})$.

lar quantities of $[\text{Me}_4\text{N}]\text{Cl}$, Ag_2ox and Ph_3SnCl in methanol. However, the synthesis yielded the expected product $[\text{Me}_4\text{N}]^+ [\text{Ph}_3\text{Snox}]^-$ as a 1/1 complex with $[(\text{Ph}_3\text{Sn})_2\text{ox}]$ instead. The structure of the quaternary-ammonium trinuclear stannate is described in this paper.

2. Experimental section

An aqueous solution containing two equivalent amounts of AgNO_3 was added to an aqueous solution containing Na_2ox to give an immediate white precipitate of Ag_2ox . The oxalate was collected, washed with water and air dried. A mixture of equimolar amounts of Ag_2ox , Ph_3SnCl and Me_4NCl in a small volume of methanol was stirred for several hours. The mixture was filtered and the solvent was allowed to evaporate slowly. A white solid was obtained, and was recrystallized from ethanol to yield large transparent plates of $[\text{Me}_4\text{N}]^+ [(\text{Ph}_3\text{Sn})_3\text{ox}_2]^-$. This compound begins to melt at about 120°C, but is not completely liquefied even at 200°C.

The crystal used in the diffraction measurements measured about $0.14 \times 0.28 \times 0.28 \text{ mm}^3$. Unit-cell dimensions were calculated from the 25 most intense reflections in the $\theta = 9.5\text{--}12.5^\circ$ range. Intensity data were collected with the Enraf-Nonius CAD4 instrument operating in the $\omega\text{-}2\theta$ scan mode up to $2\theta_{\text{max}} = 45^\circ$ (collection range: $0 \leq h \leq 10$, $0 \leq k \leq 28$, $-23 \leq l \leq 23$). The data set consisted of 8131 reflections; of the 6882 unique data 5094 data satisfied the $I \geq 3\sigma(I)$ criterion. Peak profiles were calculated [8]. The data were corrected for decay of 12.5% over 108 h of exposure to X-rays, and for absorption effects [9]. The positions of the three Sn atoms were derived by direct

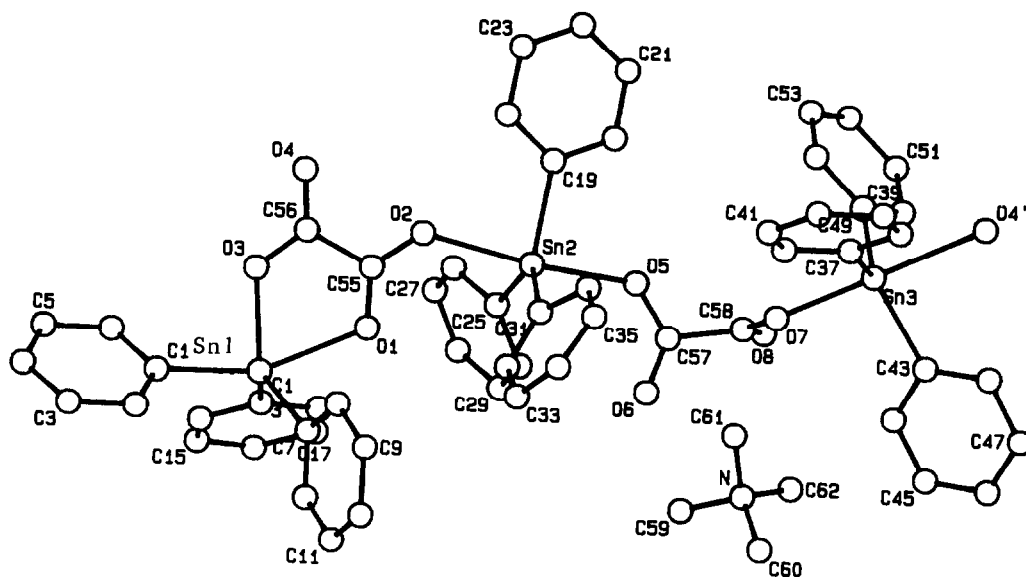


Fig. 1. Atom labelling for the asymmetric unit of $[\text{Me}_4\text{N}]^+ [(\text{Ph}_3\text{Sn})_3\text{ox}_2]^-$. Hydrogen atoms not shown. Symmetry transformation ($'$): $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$.

phase determination. Subsequent difference Fourier syntheses yielded the non-H atoms. Non-H atoms were refined anisotropically; refinement on F gave a final R index of 0.041 for the 667 variables; shift/error $_{\text{max}} \leq 0.01$; $R_w = 0.045$ with $w = [\sigma^2(F) + (0.02F)^2 + 1]^{-1}$ [10]. H-atoms were placed at calculated positions ($\text{C}-\text{H} = 0.95 \text{ \AA}$, $B = 5 \text{ \AA}^2$) in the structure-factor calculations. The final difference map had peaks of $-0.13(4)$ to $0.56(4) \text{ e\AA}^{-3}$. Scattering factors and anomalous dispersion coefficients were taken from the *International Tables for X-ray Crystallography*, Vol. IV (1974). Computations were performed with the MOLEN package [11] on a MicroVAX minicomputer. Atomic coordinates are listed in Table 1. The atomic labelling scheme is shown in Fig. 1. Additional data have been deposited with the Cambridge Crystallographic Data Centre.

2.1. Crystal data

$\text{C}_{62}\text{H}_{57}\text{NO}_8\text{Sn}_3$, FW = 1300.22, monoclinic, $P2_1/n$ (non-standard No. 14), $a = 9.777(2)$, $b = 26.679(2)$, $c = 21.937(3) \text{ \AA}$, $\beta = 96.448(8)^\circ$, $V = 5685(1) \text{ \AA}^3$, $F(000) = 2600$, $D_x = 1.519 \text{ g cm}^{-3}$, $\mu = 13.65 \text{ cm}^{-1}$ for $Z = 4$.

3. Results and discussion

The reaction of equimolar amounts of Ph_3SnCl , Ag_2ox and Me_4NCl in methanol gave $[\text{Me}_4\text{N}]^+ [(\text{Ph}_3\text{Sn})_3\text{ox}_2]^-$. The trinuclear anionic unit consists of a *cis*-tbp $[\text{Ph}_3\text{Snox}]^-$ anion ($\text{Sn1}-\text{O3}_{\text{ax}} = 2.352(5)$, $\text{Sn1}-\text{O1}_{\text{eq}} = 2.113(5) \text{ \AA}$; $\text{O1}-\text{Sn1}-\text{O3} = 71.1(2)^\circ$) (Fig. 2) that is coordinated ($\text{Sn2} \leftarrow \text{O2} = 2.322(5) \text{ \AA}$) axially to the $[(\text{Ph}_3\text{Sn})_2\text{ox}]$ ($\text{Sn2}-\text{O5} = 2.187(6) \text{ \AA}$, $\Sigma\text{C}-\text{Sn2}-\text{C} = 359.8(9)^\circ$, $\text{O2}-\text{Sn2}-\text{O5} = 174.2(2)^\circ$) molecule (Fig. 3)

through its oxalato O2 end. The oxalato group in the *cis*-tbp anion is also engaged in coordination through its O4 end ($\text{Sn3}' \leftarrow \text{O4} = 2.451(5) \text{ \AA}$) to an adjacent $[(\text{Ph}_3\text{Sn})_2\text{ox}]$ ($\text{Sn3}-\text{O7} = 2.138(5) \text{ \AA}$, $\Sigma\text{C}-\text{Sn3}-\text{C} = 359.1(9)$, $\text{O4}'-\text{Sn3}-\text{O7} = 179.6(2)^\circ$) molecule. Arising from the tetradentate bonding mode of the oxalato group belonging to the $[\text{Ph}_3\text{Snox}]^-$ species, a helical

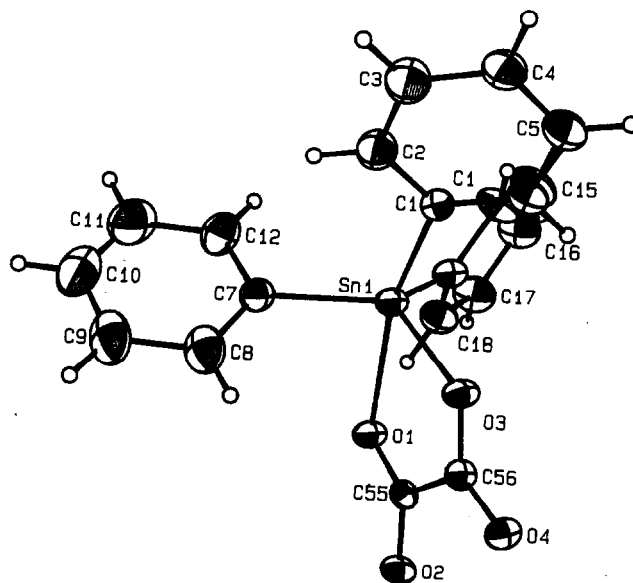


Fig. 2. Geometry of the Sn atom in the $[\text{Ph}_3\text{Snox}]^-$ anion. Selected bond distances and angles: $\text{Sn1}-\text{O1} = 2.352(5)$, $\text{Sn1}-\text{O3} = 2.113(5)$, $\text{Sn1}-\text{C1} = 2.159(9)$, $\text{Sn1}-\text{C7} = 2.11(1)$, $\text{Sn1}-\text{C13} = 2.126(9) \text{ \AA}$; $\text{O1}-\text{Sn1}-\text{O3} = 71.1(2)$, $\text{O1}-\text{Sn1}-\text{C1} = 156.0(3)$, $\text{O1}-\text{Sn1}-\text{C7} = 84.7(3)$, $\text{O1}-\text{Sn1}-\text{C13} = 87.5(3)$, $\text{O3}-\text{Sn1}-\text{C1} = 85.2(3)$, $\text{O3}-\text{Sn1}-\text{C7} = 120.1(3)$, $\text{O3}-\text{Sn1}-\text{C13} = 114.2(3)$, $\text{C1}-\text{Sn1}-\text{C7} = 104.8(4)$, $\text{C1}-\text{Sn1}-\text{C13} = 106.1(3)$, $\text{C7}-\text{Sn1}-\text{C13} = 118.7(4)^\circ$.

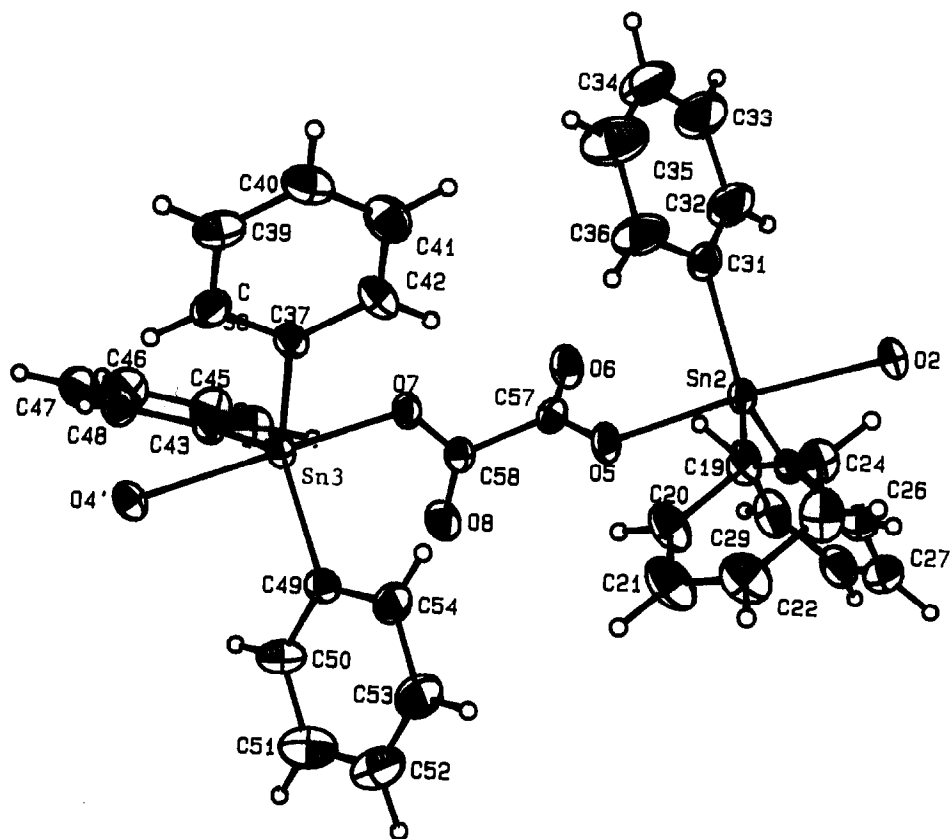


Fig. 3. Geometry of Sn atoms in the $[(\text{Ph}_3\text{Sn})_2\text{ox}]$ molecule. Selected bond distances and angles: Sn2–O2 = 2.322(5), Sn2–O5 = 2.187(6), Sn2–C19 = 2.141(8), Sn2–C25 = 2.117(8), Sn2–C31 = 2.102(9), Sn3–O4' = 2.451(5), Sn3–O7 = 2.138(5), Sn3–C37 = 2.147(9), Sn3–C43 = 2.142(8), Sn3–C49 = 2.120(8) Å; O2–Sn2–O5 = 174.1(2), O2–Sn2–C19 = 85.8(3), O2–Sn2–C25 = 88.4(3), O2–Sn2–C31 = 90.6(3), O5–Sn2–C19 = 88.6(3), O5–Sn2–C25 = 92.4(3), O5–Sn2–C31 = 93.6(3), C19–Sn2–C25 = 116.5(3), C19–Sn2–C31 = 115.5(3), C25–Sn2–C31 = 127.8(3), O4'–Sn3–O7 = 179.6(2), O4'–Sn3–C37 = 91.7(3), O4'–Sn3–C43 = 84.5(3), O4'–Sn3–C49 = 84.0(3), O7–Sn3–C37 = 88.3(3), O7–Sn3–C43 = 95.8(3), O7–Sn3–C49 = 95.7(3), C37–Sn3–C43 = 118.1(3), C37–Sn3–C49 = 120.6(3), C43–Sn3–C49 = 120.4(3)°. Symmetry transformation ('): $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$.

$[\text{Ph}_3\text{Snox} \cdot (\text{Ph}_3\text{Sn})_2\text{ox}]_n^{n-}$ chain running parallel to the *b*-axis is generated. In the $[(\text{Ph}_3\text{Sn})_2\text{ox}]$ molecule the oxalato group behaves merely as a bridge to link together two Ph_3Sn planar cations, but whereas the tetradentate oxalato group is almost flat, the bridging oxalato group is twisted so that the two CO_2 entities are nearly perpendicular to each other.

Few examples of *cis*-tbp geometries [12] are found in the structural literature of triorganotin compounds. The structure of the $[\text{Ph}_3\text{Snox}]^-$ anion in the present trinuclear stannate is similar to that in $[\text{Ph}_3\text{SnON}(\text{Ph})\text{C}(\text{O})\text{Ph}]$ [13] in that both compounds possess a five-membered chelate ring. In the hydroxamate the benzoyl O atom occupies the axial position of the *cis*-tbp, so that the Sn–O_{ax} bond distance (2.308(4) Å) exceeds the Sn–O_{eq} bond distance (2.091(5) Å). The bite angle is only 71.3(2)°. A relatively long C–O bond (1.265(9) Å) implies delocalization of the positive charge on to the N atom. A larger chelate bite should cause less distortion of the geometry; for $[\text{Ph}_3\text{SnOC}(\text{Ph})\text{CHCO-Ph}]$ which has a six-membered chelate ring (Sn–O_{ax} =

2.276(7), Sn–O_{eq} = 2.094(7) Å) the bite is increased to 78.1(2)° [14], so that the axial O atom can approach the Sn atom at a closer range.

In the $[\text{Ph}_3\text{Snox}]^-$ portion of the title stannate, the Sn–O_{ax} bond (Sn1–O1 2.352(5) Å) is much longer than the Sn–O_{eq} bond (Sn1–O3 = 2.113(5) Å). The bite angle (71.1(2)°) is comparable with those found in other chelated oxalato-tin systems [5–7]. The axial Sn–C bond (Sn1–C1 = 2.159(9) Å) is somewhat longer than the equatorial ones (Sn1–C7 = 2.11(1), Sn1–C13 = 2.126(9) Å). The axial skeleton is severely bent, (O1–Sn1–C1 = 156.0(3) Å), and the Sn atom is displaced out of the equatorial plane ($\Sigma_{\text{angles at Sn}} = 353(1)^\circ$) in the direction of the apical C1 atom by 0.328(1) Å.

The oxalato group in the $[\text{Ph}_3\text{Snox}]^-$ ion consists of two dissimilar $-\text{CO}_2$ entities. One (O1–C55–O2) binds axially to the Sn1 atom and has almost equivalent C–O bonds (C–O = 1.242(9), 1.253(9) Å), so that the negative charge can be considered to have been delocalized over the three atoms. The axial carboxyl O2 end bears

a partial negative charge which enables it to approach the Sn2 atom of the $[(\text{Ph}_3\text{Sn})_2\text{ox}]$ molecule at a relatively short distance of 2.322(5) Å. On the other hand, the $-\text{CO}_2$ half connected equatorially has bonds (C–O = 1.280(9), 1.229(9) Å) that can be unambiguously distinguished as single and double bonds. The equatorial carboxyl O4 end bears no nett charge, and can only approach the Sn3 atom of the adjacent $[(\text{Ph}_3\text{Sn})_2\text{ox}]$ molecule at a much longer (2.451(5) Å) distance.

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