listed in Table 1* and selected bond distances and angles in Table 2. The molecule is depicted in Fig. 1.

Related literature. The refinement is an improvement of the reported refinement for which $R = 0.048$ for 2388 $I \geq 3\sigma(I)$ data (Harrison, King & Healy, 1979).

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* Lists of structure factors, anisotropic thermal parameters and calculated H-atom positional parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55636 (28 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: OH1001]

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Structure of Bis(triphenyltin) Succinate–[Bis(triphenyltin) Succinate Bis(*N*,*N*-dimethylformamide)] (1/1)

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Abstract. $[Sn_4(C_4H_4O_4)_2(C_3H_7NO)_2(C_6H_5)_{12}]$, $M_r = 1778.38$, triclinic, $P\bar{1}$, $a = 12.1759$ (7), $b = 12.8661$ (9), $c = 14.3293$ (9) Å, $\alpha = 79.041$ (5), $\beta = 71.665$ (5), $\gamma = 69.467$ (5)°, $V = 1987.4$ (2) Å^3, $Z = 1$, $D_x = 1.486$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 13.03$ cm^{-1}, $F(000) = 892$, $T = 298$ K, $R = 0.026$ for 5135 reflections. The carbonyl O atoms of the centrosymmetric bis(triphenyltin) succinate.2DMF [Sn—O = 2.145 (3), $Sn \leftarrow O_{DMF}$ = 2.404 (3) Å] molecule are linked datively to the Sn atoms [Sn—O = 2.444 (2) Å] of two centrosymmetric bis(triphenyltin) succinate [Sn—O = 2.112 (3) Å] molecules to form a three-dimensional network. The coordination environment at each of the Sn atoms is a *trans*-trigonal bipyramid.

Experimental. The compound, m.p. 430 K, was obtained as an adventitious product on reacting $(C_6H_5)_3SnOH$ with $(CH_3)_2NCS_2CH_2CO_2H$ in hot ethanol. As the condensation performed in an earlier attempt yielded the stannyl ester $(CH_3)_2NCS_2CH_2CO_2Sn(C_6H_5)_3$ (Ng & Kumar Das, 1991), the anomalous occurrence of the $—O_2CCH_2CH_2CO_2—$ and $(CH_3)_2NCHO$ components in the present compound must be attributed to a transformation of either the parent acid or its stannyl salt, induced by an undefined impurity in the solvent used in the experiment. A parallel can be drawn to the well documented susceptibility of a number of ethylenebisdithiocarbamate salts to oxidation and hydrolysis. Aeration of a suspension of manganese ethylene-1,2-bisdithiocarbamate at pH 6, for example, results in the formation of the break-down products, ethylenethiourea, 5,6-dihydro-3*H*-imidazo-1,2,4-dithiazole-3-thione, ethylenediamine, carbon disulfide and elemental sulfur (Vonk, 1975).

A crystal measuring approximately 0.11 × 0.18 × 0.22 mm was used in the study. Cell dimensions were calculated from the 25 most intense reflections in the $13 \leq \theta \leq 15°$ shell. Data were collected on an Enraf–Nonius CAD-4 diffractometer using ω–2θ scans to $2\theta_{max} = 50°$ (h 0 to 14, k −15 to 15, l −17 to 17). Of 7350 data measured, 6622 were unique with 5135 obeying $I \geq 3\sigma(I)$. Crystal decay was monitored by three reflections ($2\bar{4}7$, $27\bar{3}$ and $45\bar{5}$). A linear decay correction was applied owing to a decrease in their intensities of 6.5% during the 61 h of data collection (minimum/maximum corrections 1.00002/1.03396;

Table 1. *Positional parameters and equivalent isotropic thermal parameters* (Å^2)

$$B_{eq} = (4/3)[a^2B_{11} + b^2B_{22} + c^2B_{33} + ab(\cos\gamma)B_{12} + ac(\cos\beta)B_{13} + bc(\cos\alpha)B_{23}].$$

	x	y	z	B_{eq}
Sn1*a*	0.22136 (2)	0.29070 (2)	0.17503 (2)	2.808 (6)
O1*a*	0.3379 (2)	0.1656 (2)	0.0764 (2)	3.24 (6)
O2*a*	0.3422 (2)	0.0364 (2)	0.2028 (2)	2.82 (6)
C1*a*	0.3544 (3)	0.2773 (3)	0.2468 (3)	3.37 (9)
C2*a*	0.4596 (4)	0.3009 (4)	0.1932 (4)	4.9 (1)
C3*a*	0.5445 (4)	0.2992 (4)	0.2401 (5)	6.7 (2)
C4*a*	0.5241 (5)	0.2743 (5)	0.3387 (4)	7.1 (1)
C5*a*	0.4213 (4)	0.2501 (4)	0.3930 (4)	5.9 (1)
C6*a*	0.3369 (4)	0.2509 (4)	0.3479 (3)	4.6 (1)
C7*a*	0.2076 (3)	0.4162 (3)	0.0546 (3)	3.28 (9)
C8*a*	0.1084 (4)	0.5105 (4)	0.0620 (3)	4.7 (1)
C9*a*	0.0954 (4)	0.5892 (4)	−0.0171 (4)	5.3 (1)
C10*a*	0.1802 (5)	0.5754 (4)	−0.1056 (4)	5.8 (1)
C11*a*	0.2771 (6)	0.4832 (5)	−0.1132 (5)	9.2 (2)
C12*a*	0.2913 (5)	0.4038 (4)	−0.0336 (4)	7.6 (2)
C13*a*	0.0726 (3)	0.2262 (3)	0.2224 (3)	3.11 (9)
C14*a*	−0.0184 (4)	0.2468 (4)	0.3095 (3)	4.8 (1)
C15*a*	−0.1168 (4)	0.2051 (5)	0.3324 (4)	6.3 (2)
C16*a*	−0.1236 (5)	0.1464 (4)	0.2671 (4)	6.4 (1)
C17*a*	−0.0368 (4)	0.1262 (4)	0.1808 (4)	6.1 (1)
C18*a*	0.0609 (4)	0.1651 (3)	0.1587 (3)	4.2 (1)
C19*a*	0.3724 (3)	0.0647 (3)	0.1132 (3)	2.51 (8)
C20*a*	0.4514 (3)	−0.0197 (3)	0.0391 (3)	2.99 (9)
O1	0.0939 (3)	0.4332 (2)	0.2841 (2)	4.86 (8)
C2	0.1280 (5)	0.4941 (4)	0.3173 (4)	4.9 (1)
N3	0.0556 (4)	0.5784 (3)	0.3683 (3)	5.6 (1)
C4	0.1037 (6)	0.6461 (5)	0.4046 (5)	8.9 (2)
C5	−0.0741 (6)	0.6078 (5)	0.3853 (6)	9.2 (2)
Sn1*b*	0.41792 (2)	−0.13753 (2)	0.30005 (2)	2.974 (6)
O1*b*	0.5069 (3)	−0.2887 (2)	0.2712 (2)	4.79 (8)
O2*b*	0.3393 (3)	−0.3281 (3)	0.4588 (3)	5.6 (1)
C1*b*	0.3071 (4)	−0.0416 (3)	0.4198 (3)	3.27 (9)
C2*b*	0.1843 (4)	0.0093 (4)	0.4324 (3)	4.2 (1)
C3*b*	0.1127 (5)	0.1127 (5)	0.0711 (4)	5.4 (1)
C4*b*	0.1644 (5)	0.0829 (4)	0.5784 (4)	5.5 (1)
C5*b*	0.2864 (5)	0.0355 (4)	0.5672 (3)	5.7 (1)
C6*b*	0.3567 (4)	−0.0270 (4)	0.4888 (3)	4.6 (1)
C7*b*	0.5934 (3)	−0.1163 (3)	0.2308 (3)	3.34 (9)
C8*b*	0.6087 (4)	−0.0121 (4)	0.2070 (3)	4.2 (1)
C9*b*	0.7207 (4)	0.0004 (5)	0.1570 (4)	5.7 (1)
C10*b*	0.8189 (5)	−0.0904 (5)	0.1310 (4)	7.2 (2)
C11*b*	0.8074 (5)	−0.1932 (6)	0.1557 (5)	7.8 (2)
C12*b*	0.6950 (5)	−0.2069 (4)	0.2063 (4)	5.9 (2)
C13*b*	0.3391 (4)	−0.2076 (3)	0.2244 (3)	3.9 (1)
C14*b*	0.3944 (5)	−0.3161 (4)	0.2004 (4)	5.6 (1)
C15*b*	0.3494 (6)	−0.3601 (5)	0.1464 (5)	9.1 (2)
C16*b*	0.2492 (6)	−0.3004 (5)	0.1178 (5)	11.4 (2)
C17*b*	0.1903 (5)	−0.1938 (5)	0.1429 (5)	9.7 (2)
C18*b*	0.2355 (4)	−0.1463 (4)	0.1943 (4)	6.2 (1)
C19*b*	0.4491 (4)	−0.3531 (3)	0.4303 (3)	4.0 (1)
C20*b*	0.5320 (4)	−0.4636 (3)	0.4622 (4)	4.9 (1)

Table 2. *Selected bond distances* (Å) *and angles* (°)

Sn1*a*—O1*a*	2.145 (3)	Sn1*b*—O2*a*	2.444 (2)
Sn1*a*—C1*a*	2.120 (4)	Sn1*b*—O1*b*	2.112 (3)
Sn1*a*—C7*a*	2.132 (4)	Sn1*b*—C1*b*	2.114 (4)
Sn1*a*—C13*a*	2.121 (4)	Sn11*b*—C7*b*	2.144 (4)
Sn1*a*—O1	2.404 (3)	Sn1*b*—C13*b*	2.131 (5)
O1*a*—Sn1*a*—C1*a*	95.1 (1)	O2*a*—Sn1*b*—C1*b*	82.7 (1)
O1*a*—Sn1*a*—C7*a*	91.3 (1)	O2*a*—Sn1*b*—C7*b*	84.9 (1)
O1*a*—Sn1*a*—C13*a*	95.8 (1)	O2*a*—Sn1*b*—C13*b*	85.3 (1)
O1*a*—Sn1*a*—O1	178.7 (1)	O1*b*—Sn1*b*—C1*b*	102.5 (1)
C1*a*—Sn1*a*—C7*a*	115.7 (2)	O1*b*—Sn1*b*—C7*b*	87.6 (1)
C1*a*—Sn1*a*—C13*a*	129.8 (2)	O1*b*—Sn1*b*—C13*b*	96.8 (2)
C1*a*—Sn1*a*—O1	84.2 (1)	C1*b*—Sn1*b*—C7*b*	116.5 (2)
C7*a*—Sn1*a*—C13*a*	112.8 (2)	C1*b*—Sn1*b*—C13*b*	120.3 (2)
C7*a*—Sn1*a*—O1	88.1 (1)	C7*b*—Sn1*b*—C13*b*	120.3 (2)
C13*a*—Sn1*a*—O1	85.4 (1)	Sn1*b*—O2*a*—C19*a*	131.4 (2)
Sn1*a*—O1*a*—C19*a*	118.1 (2)	Sn1*b*—O1*b*—C19*b*	122.5 (3)
O2*a*—Sn1*b*—O1*b*	172.2 (1)		

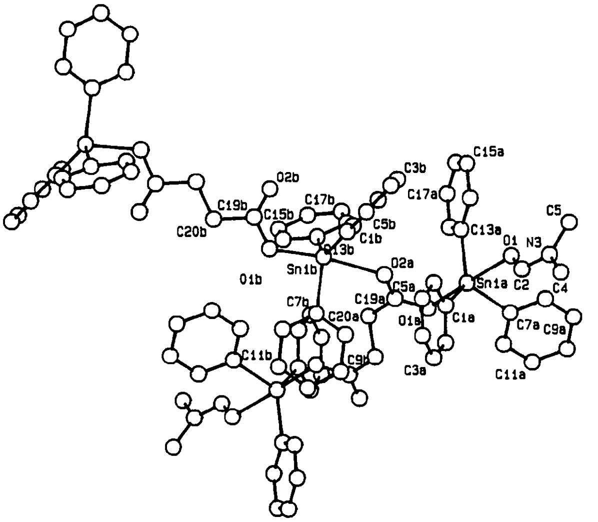

Fig. 1. Plot of the tetranuclear bis(triphenyltin) succinate–[bis(triphenyltin) succinate bis-*N*,*N*-dimethylformamide] complex. The center of inversion lies midway between the methylene C atoms of the succinato anion.

average correction 1.01533). The data set was corrected for absorption by using ψ-scan data (minimum/maximum transmission 0.9608/0.9998; average transmission 0.9804). The structure was solved by the heavy-atom method, and all non-H atoms were refined anisotropically. H atoms were generated (C—H = 0.95 Å, $B = 5$ Å^2) and were allowed to ride on their respective C atoms. Full-matrix least-squares refinement based on F for the 460 variables gave final $R = 0.026$, $wR = 0.029$ $\{w = [\sigma(F)^2 + (0.02F)^2 + 1]^{-1}\}$; $S = 0.358$; $(\Delta/\sigma)_{max} \leq 0.01$; $(\Delta\rho)_{min/max} = -0.10\ (5)/0.30\ (5)$ e Å^{-3}. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.3.1 and 2.28). Computations were performed by using the *MolEN* structure determination package (Fair, 1990) on a DEC MicroVAX minicomputer. Fractional atomic coordinates are listed in Table 1;* selected bond distances and angles are listed in Table 2. The tetranuclear complex is depicted in Fig. 1.

Related literature. Triphenyltin alkanoates generally do not form isolable adducts with neutral *O*-donor ligands as they themselves are coordinately saturated five-coordinate carboxylate-bridged compounds (Tiekink, 1991). In the few reported complexes, the Sn←O bonds are typically long, as has been found,

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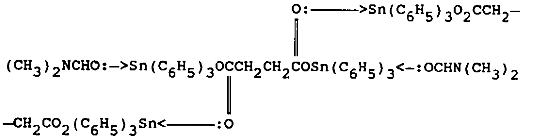
* Lists of structure factors, anisotropic thermal parameters, calculated H-atom positional parameters, and complete bond lengths and bond angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55637 (59 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: OH1002]

for example, in the quinolyloxyacetate hydrate [Sn←O = 2.338 (7), 2.391 (6) Å (Kumar Das, Chen, Ng & Mak, 1987)] and trichloroacetate.methanol [Sn←O = 2.400 (3) Å (Alcock & Roe, 1989)] complexes. The present tetranuclear complex (I) formally consists of two coordinated DMF and two independent bis(triphenyltin) succinate molecules, *a* and *b*.



$$\begin{array}{l} \qquad\qquad\qquad\qquad\quad O{:}\longrightarrow Sn(C_6H_5)_3O_2CCH_2- \\ (CH_3)_2NCHO{:}\rightarrow Sn(C_6H_5)_3OCCH_2CH_2COSn(C_6H_5)_3\leftarrow{:}OCHN(CH_3)_2 \\ -CH_2CO_2(C_6H_5)_3Sn\longleftarrow{:}O \end{array}$$

(I)

In the complex, the Sn1*a*←O$_{DMF}$ bond distance is only slightly shorter than the Sn←O carboxylate bridge and compares well with the 2.402 (5) Å length found in the DMF adduct of the *N*-triphenylstannyl derivative of saccharin (Ng, Kuthubutheen, Zainudin, Chen, Kumar Das, Schulze, Molloy, Yip & Mak, 1991). The intermolecular bridging distance of 2.444 (2) Å is longer than that found in a monocarboxylate such as triphenyltin acetate [Sn←O = 2.349 (3) Å (Molloy, Purcell, Quill & Nowell, 1984)], but is similar to those found in the bis(trimethyltin) derivatives of acetylenedicarboxylic [Sn←O = 2.401 (4), 2.408 (4) Å], terephthalic [Sn←O = 2.506 (3) Å] (Glowacki, Huber & Preut, 1988) and malonic [Sn←O = 2.44 (2), 2.46 (2) Å (Schubert, 1978)] acids.

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Bis(tetrabutylammonium) Nonadecaoxohexamolybdenum(VI): a Second Polymorph

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Abstract. $[C_{16}H_{36}N]_2[Mo_6O_{19}]$, $M_r = 1364.6$, monoclinic, $C2/c$, $a = 16.280\,(3)$, $b = 17.204\,(3)$, $c = 17.738\,(4)$ Å, $\beta = 101.42\,(2)°$, $V = 4869.3\,(17)$ Å^3, $Z = 4$, $D_x = 1.861$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 15.66$ cm^{-1}, $F(000) = 2728$, $T = 298$ K, $R = 0.0633$ for 2857 observed reflections and 269 least-squares parameters. The six Mo atoms form the standard distorted octahedral cage with O atoms bridging the Mo atoms. There is a crystallographic twofold axis in the anionic cluster ($Mo_6O_{19}^{2-}$) through the central O atom and containing Mo(2) and Mo(4).

Experimental. Orange–red crystals were grown from CH_3CN/Et_2O (0.26 × 0.32 × 0.32 mm). Data were collected on a Siemens *P*4 diffractometer with graphite monochromator, using ω scans. Lattice parameters were determined from least-squares fit of 25 reflections ($20 \leq 2\theta \leq 25°$), minimized on *F*. Semi-empirical absorption correction was applied to the data set [*XEMP* (Sheldrick, 1990), T_{max}/T_{min} = 0.178/0.092, $\mu = 15.66$ cm^{-1}]. $2\theta_{max} = 50°$ ($h = \pm 20$, $k = +21$, $l = +22$). Three standard reflections ($\bar{9}13$, $\bar{1}90$ and 0,0,10) for 4609 reflections collected, showed