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Bis(triphenyltin) Succinate and its Complex with Dimethyl Sulfoxide and Ethanol, and its Complex with Hexamethylphosphoramide

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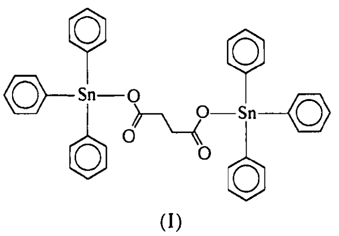
Abstract

Bis(triphenyltin) succinate exists as a centrosymmetric dinuclear molecule, μ-succinato-bis(triphenyltin), $[\mathrm{Sn_2(C_4H_4O_4)(C_6H_5)_6}]$, with its Sn atom in tetrahedral coordination. When crystallized from an ethanol solution containing excess dimethyl sulfoxide (DMSO), it yields [μ-succinato-bis(triphenyltin)].[μ-succinato-bis(triphenyltin).sesqui(dimethyl sulfoxide).hemiethanol], $[\mathrm{Sn_4(C_4H_4O_4)_2(C_6H_5)_{12}(C_2H_6O)_{1/2}(C_2H_6OS)_{3/2}}]$, and when crystallized from an ethanol solution containing excess hexamethylphosphoramide (HMPA), it yields [μ-succinato-bis(triphenyltin)].[μ-succinato-bis(triphenyltin).bis(hexamethylphosphoramide)], $[\mathrm{Sn_4(C_4H_4O_4)_2(C_6H_5)_{12}(C_6H_{18}N_3OP)_2}]$. In the DMSO and HMPA complexes, the [μ-succinato-bis(triphenyltin)] moiety is linked to the [μ-succinato-bis(triphenyltin).bis(donor)] moiety by a carboxylate bridge, giving rise to a linear chain structure of tetranuclear complexes.

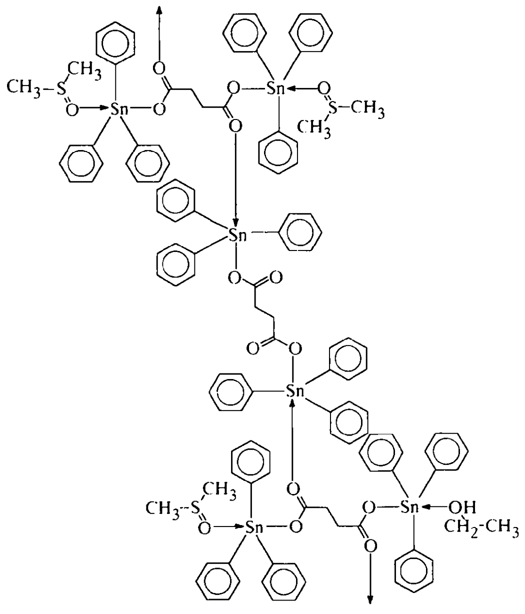
Comment

Bis(triphenyltin) succinate, (I), has been assigned tetrahedral geometry on the basis of spectroscopic measurements (Samuel-Lewis *et al.*, 1992). The compound forms a 1/1 adduct with *N*,*N*-dimethylformamide (DMF); this adduct is formally the tetranuclear complex [μ-succinato-bis(triphenyltin)].[bis(triphenyltin) succinate.2DMF] (Ng & Kumar Das, 1993). The compound affords a similar tetranuclear complex with ethanol, but this complex crystallizes with two lattice ethanol molecules (Ng *et al.*, 1994). On the other hand, with quinoline *N*-oxide as the donor ligand, the resulting hexanuclear complex contains two molecules of bis(triphenyltin) succinate.



(I)

The DMF, DMSO and HMPA complexes form linear chains in which the [μ-succinato-bis(triphenyltin)] moiety is linked to the [μ-succinato-bis(triphenyltin).bis(donor)] moiety by carboxylate bridges. The Lewis basicity of the donor (HMPA $\gg$ DMSO $>$ DMF) exerts no effect on the overall architecture of the complex. In the DMSO complex, (II), the latter moiety contains only 1.5 DMSO and 0.5 ethanol; however, the 0.5 ethanol does not participate in hydrogen bonding, unlike the coordinated ethanol molecules in [μ-succinato-bis(triphenyltin)].[μ-succinato-bis(triphenyltin).2ethanol].2ethanol (Ng & Kumar Das, 1994).



(II)

The covalent Sn—O distance in the parent Lewis acid (I) [2.058 (2) Å] is similar to distances found in tetrahedral triphenyltin benzoates (Tiekink, 1991, 1994). Bond dimensions involving the μ-succinato-bis(triphenyltin) moiety in complexes (II) and (III) are similar to those found in five-coordinate carboxylate-bridged triorganotin monocarboxylates (Ng *et al.*, 1989).

The Sn—O distance [2.404 (3) Å] in the DMF complex (Ng & Kumar Das, 1993) is similar to those in the DMSO complex (II) [2.395 (4) and 2.425 (3) Å], but is much longer than that in the HMPA complex (III) [2.288 (3) Å]. The short Sn—O distance is attributed to the fact that HMPA is a much stronger Lewis base than either DMSO or DMF. The Sn—$\mathrm{O_{HMPA}}$ distance is shorter than that [2.387 (3) Å] found in triphenyltin bis(*N*,*N*-dimethyldithiocarbamoyl)acetate.HMPA (Ng, 1995).

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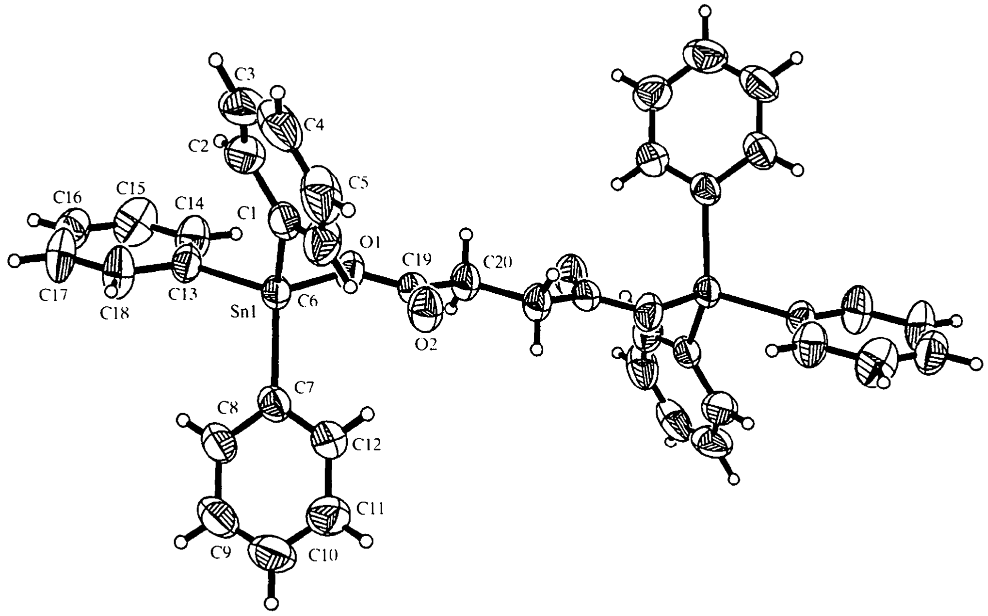
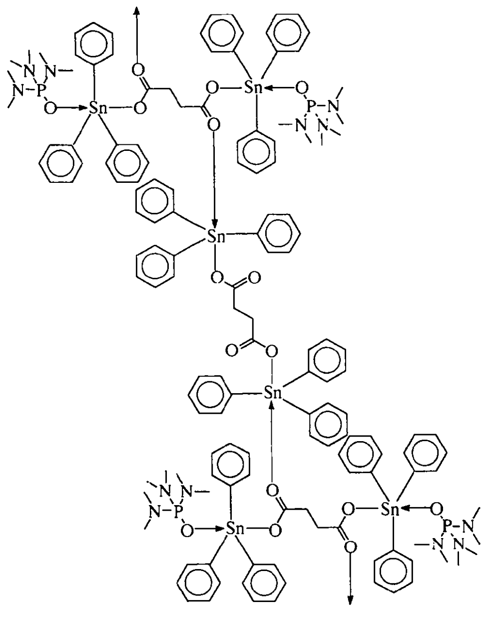


Fig. 1. Plot of (I) drawn at the 25% probability level. H atoms are shown as spheres of arbitrary radii.



(III)

In solution, the DMSO complex dissociates into species having the Sn atoms in tetrahedral coordination, as evidenced by the magnitude of the one-bond ^{13}C–^{119}Sn coupling constant of 652.9 Hz. This value falls within the range found for four-coordinate triphenyltin carboxylates (Holeček *et al.*, 1983).

Experimental

Bis(triphenyltin) succinate, (I), was synthesized by condensing triphenyltin hydroxide with succinic acid; the reagents (in a 2:1 molar ratio) were heated in a small volume of ethyl acetate until they dissolved completely. Slow cooling of the filtered solution yielded large crystals of the dinuclear compound (I). The condensation was then performed with ethanol as the solvent. When the reactants had dissolved, several drops of dimethyl sulfoxide were added to the solution. Slow cooling of the filtered solution gave the tetranuclear complex (II). The half molecule of ethanol in the formulation was suggested by the ^1H NMR spectrum recorded in $CDCl_3$. ^{13}C NMR chemical shifts in p.p.m. (^{13}C–^{119}Sn coupling constants in parentheses) in $CDCl_3$: C_{ipso} 138.24 (652.9 Hz), C_{ortho} 136.68 (47.7 Hz), C_{meta} 128.72 (63.3 Hz), C_{para} 129.90 (13.5 Hz); $(CH_3)_2SO$ 40.85, $-CH_2-$ 30.11, C_2H_5OH 18.31, 58.11. With hexamethylphosphoramide in place of dimethyl sulfoxide, the condensation in ethanol gave (III).

Compound (I)

Crystal data

$[Sn_2(C_4H_4O_4)(C_6H_5)_6]$
$M_r = 816.05$
Monoclinic
$P2_1/c$
$a = 9.535\,(2)$ Å
$b = 19.225\,(2)$ Å
$c = 9.760\,(2)$ Å
$\beta = 102.98\,(1)^\circ$
$V = 1743.5\,(6)$ Å^3
$Z = 2$
$D_x = 1.554$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 14.5$–15.0°
$\mu = 1.472$ mm^{-1}
$T = 298\,(2)$ K
Block
0.50 × 0.43 × 0.36 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer

4043 reflections with $I > 2\sigma(I)$

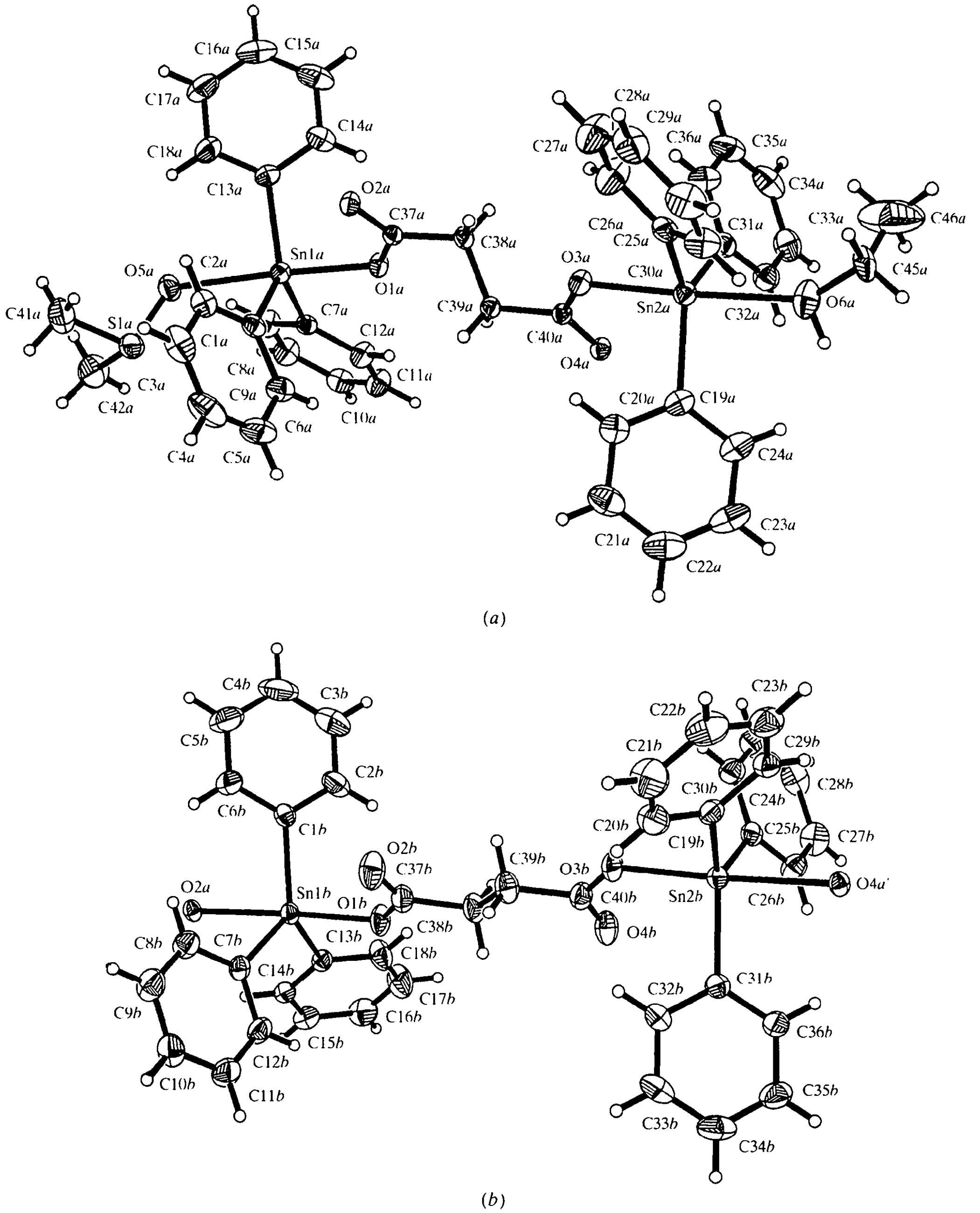


Fig. 2. (*a*) Plot of the [μ-succinato-bis(triphenyltin).sesqui(dimethyl sulfoxide).hemiethanol] moiety of (II). The ethanol is disordered with respect to another dimethyl sulfoxide molecule (not shown). (*b*) Plot of the μ-succinato-bis(triphenyltin) moiety of (II) [translational code: (′) $1+x$, $y-1$, z]. Displacement ellipsoids are drawn at the 25% probability level and H atoms are drawn as spheres of arbitrary radii.

ω scan
Absorption correction:
ψ scan (North *et al.*, 1968)
$T_{\min} = 0.496$, $T_{\max} = 0.589$
5350 measured reflections
5080 independent reflections

$R_{\rm int} = 0.030$
$\theta_{\max} = 29.97°$
$h = -13 \rightarrow 13$
$k = 0 \rightarrow 27$
$l = 0 \rightarrow 13$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.094$
$S = 1.127$
5079 reflections
208 parameters

$w = 1/[\sigma^2(F_o^2) + (0.0480P)^2 + 0.4052P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 1.098$ e Å^{-3}
$\Delta\rho_{\min} = -0.499$ e Å^{-3}

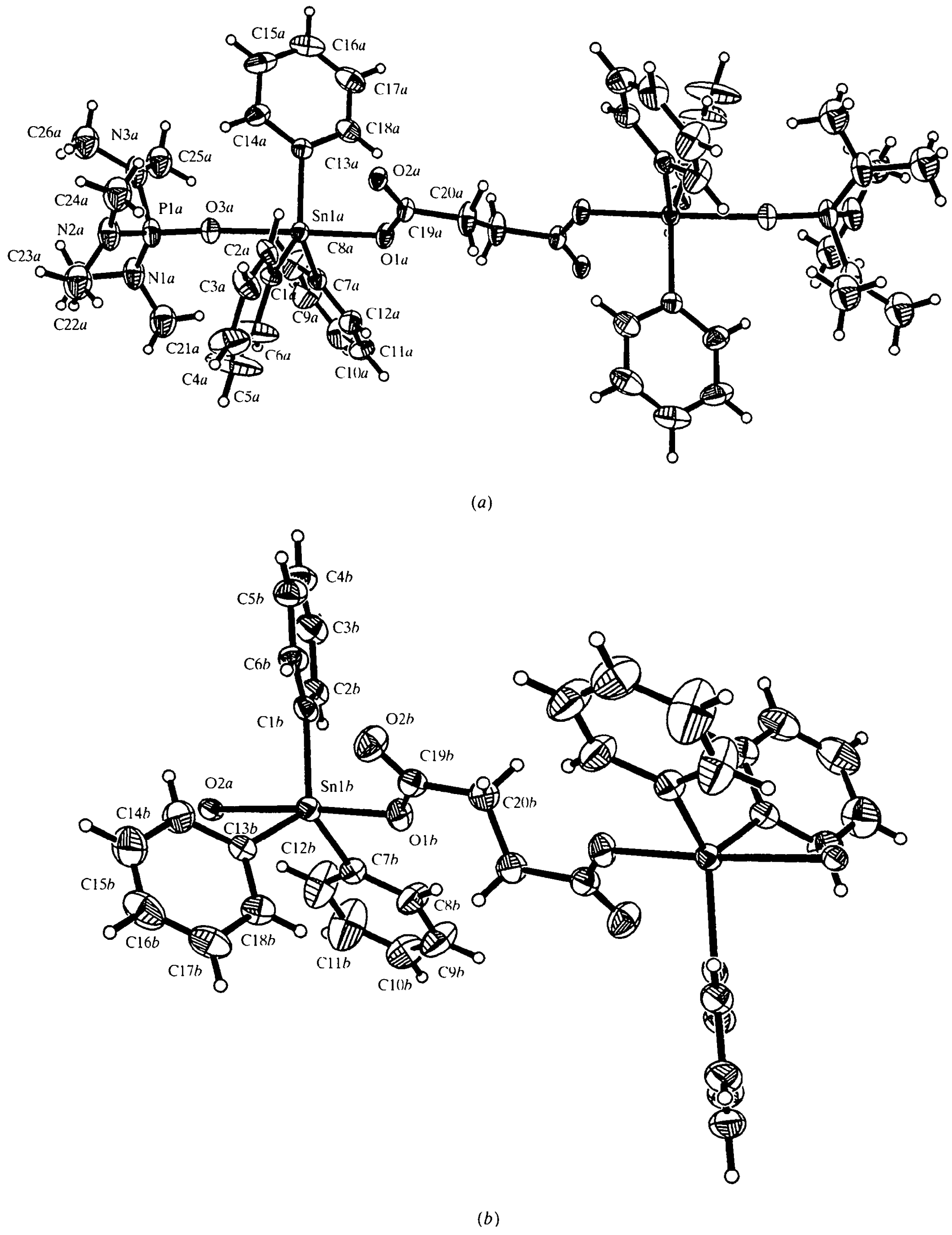


Fig. 3. (*a*) Plot of the centrosymmetric μ-succinato-bis(triphenyltin).bis(hexamethylphosphoramide) moiety of (III). (*b*) Plot of the centrosymmetric μ-succinato-bis(triphenyltin) moiety of (III). Displacement ellipsoids are drawn at the 25% probability level and H atoms are drawn as spheres of arbitrary radii.

H atoms riding, $U(H) = 1.5U_{eq}(C)$

Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

Sn1—C1	2.121 (3)	Sn1—C13	2.128 (3)
Sn1—C7	2.123 (3)	Sn1—O1	2.058 (2)

C1—Sn1—C7	116.5 (1)	C7—Sn1—C13	109.7 (1)
C1—Sn1—C13	109.4 (1)	C7—Sn1—O1	110.0 (1)
C1—Sn1—O1	112.4 (1)	C13—Sn1—O1	97.2 (1)

Compound (II)

Crystal data

$[Sn_4(C_4H_4O_4)_2(C_6H_5)_{12}$-
$(C_2H_6O)_{1/2}(C_2H_6OS)_{3/2}]$
$M_r = 1772.33$
Triclinic
$P\overline{1}$
$a = 14.206$ (2) Å
$b = 15.777$ (1) Å
$c = 20.198$ (2) Å
$\alpha = 67.565$ (6)°
$\beta = 74.546$ (8)°
$\gamma = 70.904$ (8)°
$V = 3901.7$ (6) Å^3
$Z = 2$
$D_x = 1.509$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 14.00$–$14.25°$
$\mu = 1.362$ mm^{-1}
$T = 298$ (2) K
Block
0.44 × 0.44 × 0.22 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scan
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.649$, $T_{max} = 0.741$
14 344 measured reflections
13 698 independent reflections

10 805 reflections with $I > 2\sigma(I)$
$R_{int} = 0.011$
$\theta_{max} = 24.97°$
$h = -16 \rightarrow 16$
$k = -17 \rightarrow 18$
$l = 0 \rightarrow 23$
3 standard reflections
frequency: 60 min
intensity decay: 9.8%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.101$
$S = 1.041$
13 695 reflections
775 parameters
H atoms riding, $U(H) = 1.5U_{eq}(C)$; hydroxy H atom calculated

$w = 1/[\sigma^2(F_o^2) + (0.0496P)^2 + 3.8919P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 1.554$ e Å^{-3}
$\Delta\rho_{min} = -0.669$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

Sn1*a*—C1*a*	2.123 (2)	Sn1*b*—C1*b*	2.130 (3)
Sn1*a*—C7*a*	2.143 (2)	Sn1*b*—C7*b*	2.120 (2)
Sn1*a*—C13*a*	2.126 (2)	Sn1*b*—C13*b*	2.155 (2)
Sn1*a*—O1*a*	2.154 (3)	Sn1*b*—O1*b*	2.099 (3)
Sn1*a*—O5*a*	2.425 (3)	Sn1*b*—O2*a*	2.426 (3)
Sn2*a*—C19*a*	2.133 (2)	Sn2*b*—C19*b*	2.154 (2)
Sn2*a*—C25*a*	2.137 (2)	Sn2*b*—C25*b*	2.122 (2)
Sn2*a*—C31*a*	2.129 (2)	Sn2*b*—C31*b*	2.144 (2)
Sn2*a*—O3*a*	2.154 (3)	Sn2*b*—O3*b*	2.117 (3)
Sn2*a*—O6*a*	2.395 (4)	Sn2*b*—O4*a*'	2.433 (3)
C1*a*—Sn1*a*—C7*a*	116.9 (1)	C1*b*—Sn1*b*—C7*b*	122.7 (1)
C1*a*—Sn1*a*—C13*a*	130.9 (1)	C1*b*—Sn1*b*—C13*b*	118.4 (1)
C1*a*—Sn1*a*—O1*a*	92.3 (1)	C1*b*—Sn1*b*—O1*b*	97.6 (1)
C1*a*—Sn1*a*—O5*a*	82.6 (1)	C1*b*—Sn1*b*—O2*a*	84.9 (1)
C7*a*—Sn1*a*—C13*a*	111.0 (1)	C7*b*—Sn1*b*—C13*b*	116.6 (1)
C7*a*—Sn1*a*—O1*a*	91.4 (1)	C7*b*—Sn1*b*—O1*b*	99.9 (1)
C7*a*—Sn1*a*—O5*a*	90.3 (1)	C7*b*—Sn1*b*—O2*a*	82.8 (1)
C13*a*—Sn1*a*—O1*a*	96.8 (1)	C13*b*—Sn1*b*—O1*b*	87.2 (1)
C13*a*—Sn1*a*—O5*a*	87.2 (1)	C13*b*—Sn1*b*—O2*a*	87.2 (1)
O1*a*—Sn1*a*—O5*a*	174.8 (1)	O1*b*—Sn1*b*—O2*a*	174.4 (1)
C19*a*—Sn2*a*—C31*a*	128.3 (1)	C19*b*—Sn2*b*—C25*b*	117.7 (1)
C19*a*—Sn2*a*—C25*a*	113.0 (1)	C19*b*—Sn2*b*—C31*b*	118.4 (1)
C19*a*—Sn2*a*—O3*a*	95.8 (1)	C19*b*—Sn2*b*—O3*b*	87.5 (1)
C19*a*—Sn2*a*—O6*a*	87.1 (1)	C19*b*—Sn2*b*—O4*a*'	84.1 (1)
C25*a*—Sn2*a*—C31*a*	117.5 (1)	C25*b*—Sn2*b*—C31*b*	121.2 (1)
C25*a*—Sn2*a*—O3*a*	90.4 (1)	C25*b*—Sn2*b*—O3*b*	102.2 (1)
C25*a*—Sn2*a*—O6*a*	87.5 (2)	C25*b*—Sn2*b*—O4*a*'	83.8 (1)
C31*a*—Sn2*a*—O3*a*	94.1 (1)	C31*b*—Sn2*b*—O3*b*	96.4 (1)
C31*a*—Sn2*a*—O6*a*	84.9 (1)	C31*b*—Sn2*b*—O4*a*'	85.7 (1)
O3*a*—Sn2*a*—O6*a*	177.0 (1)	O3*b*—Sn2*b*—O4*a*'	171.2 (1)

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

Compound (III)

Crystal data

$[Sn_4(C_4H_4O_4)_2(C_6H_5)_{12}$-
$(C_6H_{18}N_3OP)_2]$
$M_r = 1990.51$
Triclinic
$P\overline{1}$
$a = 12.488$ (1) Å
$b = 14.068$ (2) Å
$c = 14.182$ (4) Å
$\alpha = 72.11$ (2)°
$\beta = 81.76$ (2)°
$\gamma = 78.55$ (1)°
$V = 2314.8$ (7) Å^3
$Z = 1$
$D_x = 1.428$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 14.00$–$14.25°$
$\mu = 1.159$ mm^{-1}
$T = 298$ (2) K
Cube
0.36 × 0.36 × 0.36 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scan
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.579$, $T_{max} = 0.659$
8531 measured reflections
8120 independent reflections

6845 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 24.97°$
$h = -14 \rightarrow 14$
$k = -16 \rightarrow 16$
$l = 0 \rightarrow 16$
3 standard reflections
frequency: 60 min
intensity decay: 7.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.117$
$S = 1.060$
8120 reflections
451 parameters
H atoms riding, $U(H) = 1.5U_{eq}(C)$

$w = 1/[\sigma^2(F_o^2) + (0.0708P)^2 + 1.9846P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.385$ e Å^{-3}
$\Delta\rho_{min} = -0.426$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 3. *Selected geometric parameters (Å, °) for (III)*

Sn1*a*—C1*a*	2.121 (4)	Sn1*b*—C1*b*	2.124 (2)
Sn1*a*—C7*a*	2.142 (2)	Sn1*b*—C7*b*	2.148 (3)
Sn1*a*—C13*a*	2.126 (2)	Sn1*b*—C13*b*	2.120 (2)
Sn1*a*—O1*a*	2.170 (3)	Sn1*b*—O1*b*	2.133 (3)
Sn1*a*—O3*a*	2.288 (3)	Sn1*b*—O2*a*	2.457 (3)

C1*a*—Sn1*a*—C7*a*	115.2 (2)	C1*b*—Sn1*b*—C13*b*	120.4 (1)
C1*a*—Sn1*a*—C13*a*	132.3 (2)	C1*b*—Sn1*b*—C7*b*	117.1 (1)
C1*a*—Sn1*a*—O1*a*	94.6 (2)	C1*b*—Sn1*b*—O1*b*	101.2 (1)
C1*a*—Sn1*a*—O3*a*	84.8 (2)	C1*b*—Sn1*b*—O2*a*	81.7 (1)
C7*a*—Sn1*a*—C13*a*	111.7 (1)	C7*b*—Sn1*b*—C13*b*	119.8 (1)
C7*a*—Sn1*a*—O1*a*	89.3 (1)	C7*b*—Sn1*b*—O1*b*	88.4 (1)
C7*a*—Sn1*a*—O3*a*	89.9 (1)	C7*b*—Sn1*b*—O2*a*	87.6 (1)
C13*a*—Sn1*a*—O1*a*	93.9 (1)	C13*b*—Sn1*b*—O1*b*	96.5 (1)
C13*a*—Sn1*a*—O3*a*	87.4 (1)	C13*b*—Sn1*b*—O2*a*	84.5 (1)
O1*a*—Sn1*a*—O3*a*	178.7 (1)	O1*b*—Sn1*b*—O2*a*	175.8 (1)

The final difference map in (I) had a peak of about 1 e Å^{-3} near the Sn atom. In (II), phenyl rings were refined as rigid hexagons. The dimethyl sulfoxide coordinated to the Sn2*a* atom is disordered with respect to the ethanol, and each molecule was refined with 0.5 occupancy. The C—C distance in the ethanol molecule was fixed at 1.54 ± 0.01 Å. The final difference map also had peaks of about 1 e Å^{-3} near the Sn atoms. In (III), the three dimethylamino groups are disordered over two positions and were refined with restraints (P—N = 1.65 ± 0.01, N—C = 1.45 ± 0.01 and C···C = 2.37 Å); *EADP* (*SHELXL*93; Sheldrick, 1993) restraints were imposed on these atoms. All phenyl rings were refined as rigid hexagons. One of the phenyl rings bonded to the Sn1*a* atom is also disordered over two positions; an *EADP* restraint was also applied to that ring having 0.333 occupancy. In (II) and (III), disorder affected the refinement of the phenyl rings, some of which had C—C distances that were either too long or too short when the phenyl rings were not restrained as planar hexagons.

For all compounds, 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 structures: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*93.

I thank the National Science Council for R & D (IRPA 09-02-03-0371) for supporting this work.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SX1059). Services for accessing these data are described at the back of the journal.

References

Enraf–Nonius (1988). *CAD*-4 *VAX*/*PC Fortran System. Operator's Guide to the Enraf–Nonius CAD*-4 *Diffractometer Hardware, its Software and the Operating System.* Enraf–Nonius, Delft, The Netherlands.

Hall, S. R. & Stewart, J. M. (1990). Editors. *Xtal*3.0 *Reference Manual.* Universities of Western Australia, Australia, and Maryland, USA.

Holeček, J., Handlir, K., Nadvornik, M. & Lyčka, A. (1983). *J. Organomet. Chem.* **258**, 147–153.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Ng, S. W. (1995). *Main Group Met. Chem.* **18**, 603–610.

Ng, S. W., Chen, W. & Kumar Das, V. G. (1989). *J. Organomet. Chem.* **345**, 59–64.

Ng, S. W. & Kumar Das, V. G. (1993). *Acta Cryst.* C**49**, 754–756.

Ng, S. W., Kumar Das, V. G., Wang, R.-J. & Mak, T. C. W. (1994). *Malays. J. Sci.* **15**B, 7–11.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Samuel-Lewis, A., Smith, P. J., Aupers, J. H., Hampson, D. & Povey, D. C. (1992). *J. Organomet. Chem.* **437**, 131–144.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.

Tiekink, E. R. T. (1991). *Appl. Organomet. Chem.* **5**, 1–21.

Tiekink, E. R. T. (1994). *Trends Organomet. Chem.* **1**, 71–116.

Acta Cryst. (1998). C**54**, 750-752

$2[(C_6H_{11})_2NH_2]^+.[Sn(O_2CCH_2CH_2CO_2)(C_4H_9)_3]^-.1/2(O_2CCH_2CH_2CO_2)^{2-}.H_2O$, an Organostannate Consisting of Linear Polyanionic Tributyl(succinato)stannate Chains Hydrogen Bonded into Layers by a [Tetrakis(dicyclohexylammonium) Succinate Dihydrate] Dicationic Network

SEIK WENG NG[a] AND JAROSLAV HOLEČEK[b]

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Abstract

In bis(dicyclohexylammonium) *catena*-poly[tributylstannate-μ-succinato] hemi(succinate) monohydrate, $(C_{12}H_{24}N)_2[Sn(C_4H_4O_4)(C_4H_9)_3](C_4H_4O_4)_{1/2}.H_2O$, the polyanionic tributyl(succinato)stannate chains are hydrogen bonded into layers by a [tetrakis(dicyclohexylammonium) succinate dihydrate] dicationic network. The linear polyanionic chain consists of planar tributyltin groups that are axially linked by the succinato groups.

Comment

The dicyclohexylammonium hydrogen carboxylates, $[(C_6H_{11})_2NH_2]^+.[HO_2C(CH_2)_nCO_2]^-$, when condensed with half a molar equivalent of bis(tributyltin) oxide in ethanol, yield bis(dicyclohexylammonium) tris(oxalato)tetrakis(tributylstannate).2ethanol for the $n = 0$ (oxalato) homolog (Ng *et al.*, 1990) and bis(dicyclohexylammonium) tris(malonato)tetrakis(tributylstannate) for the $n = 1$ (malonato) derivative (Ng *et al.*, 1992). The condensation of dicyclohexylammonium hydrogen succinate ($n = 2$) with bis(tributyltin) does not afford the simple ammonium stannate, but instead gives $[(C_6H_{11})_2NH_2]^+.[Sn(C_4H_9)_3(O_2CCH_2CH_2CO_2)]^-$ as a 2:1 complex with $2[(C_6H_{11})_2NH_2]^+.[O_2CCH_2CH_2CO_2]^{2-}.2H_2O$, (I).

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