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Supplementary data for this paper are available from the IUCr electronic archives (Reference: DA1054). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). C**55**, 744–748

catena-Poly[triphenyltin-μ-(*N*,*N*-diethylthiocarbamoylthioacetato-*O*:*O*′)], *catena*-poly[triphenyltin-μ-(*N*-methyl-*N*-phenylthiocarbamoylthioacetato-*O*:*O*′)] and triphenyl(*N*,*N*-tetramethylenethiocarbamoylthioacetato-*O*)tin

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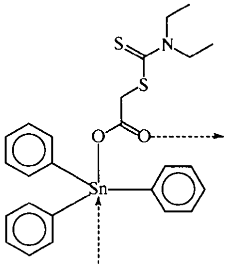
Abstract

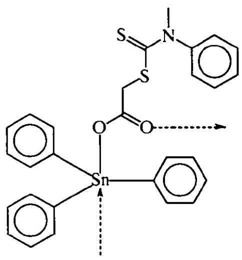
Carboxylate bridges link the two independent molecules of triphenyltin *N*,*N*-diethylthiocarbamoylthioacetate, (I), and the four independent molecules of triphenyltin *N*-methyl-*N*-phenylthiocarbamoylthioacetate, (II), into linear chains whose Sn atoms show *trans*-trigonal-bipyramidal coordination, *i.e.* (I) is *catena*-poly[triphenyltin-μ-(*N*,*N*-diethylthiocarbamoylthioacetato-*O*:*O*′)], [$Sn(C_6H_5)_3(\mu\text{-}C_7H_{12}NO_2S_2)$]$_n$, and (II) is *catena*-poly[triphenyltin-μ-(*N*-methyl-*N*-phenylthiocarbamoylthioacetato-*O*:*O*′)], [$Sn(C_6H_5)_3(\mu\text{-}C_{10}H_{10}NO_2S_2)$]$_n$. Triphenyltin *N*,*N*-tetramethylenethiocarbamoylthioacetate {or triphenyl(*N*,*N*-tetramethylenethiocarbamoylthioacetato-*O*)tin, [$Sn(C_6H_5)_3(C_7H_{10}NO_2S_2)$]} exists as a monomeric tetrahedral molecule.

Comment

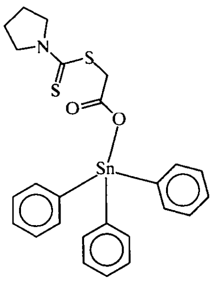
Triphenyltin alkanoates adopt linear carboxylate-bridged polymeric structures that usually propagate by twofold screw-axial translations; these polymers display a repeat distance of 5.2 Å, this distance being generally insensitive to the nature of the substituent on the carboxylate anion (Ng *et al.*, 1988). Triphenyltin *N*,*N*-dimethylthiocarbamoylthioacetate, which crystallizes with two symmetry-independent molecules in the unit cell, propagates by a twofold screw axis, but its repeat distance of 4.3 Å represents an anomaly (Ng & Kumar Das, 1995). The shorter repeat distance implies a more helical conformation of the polymeric chain. The repeat distance contrasts with a length of 4.9 Å for trimethyltin *N*,*N*-dimethylthiocarbamoylthioacetate, whose two symmetry-independent molecules propagate by mere translations, so that the architecture approaches a zigzag arrangement (Ng & Kumar Das, 1996). The rigidities of the polymeric backbone in the two carboxylates can be distinguished by the slope of the variable-temperature Sn^{119m} Mössbauer plot (Ng & Kumar Das, 1991).



(I)



(II)



(III)

The title *N*,*N*-diethylthiocarbamoylthioacetate polymer, (I), displays a three-line Mössbauer spectrum that has been interpreted in terms of two Sn sites, one in a five-coordinate environment and the other in a four-coordinate environment (Ng & Kumar Das, 1991). This assignment is, however, not supported by crystal structure analysis. The compound crystallizes with cell constants that match those of the *N*,*N*-dimethylthiocarbamoylthioacetate polymer; the two structures also show matching atomic coordinates, so that the origin of the three-line Mössbauer spectrum remains obscure. On the other hand, tricyclohexyltin *N*,*N*-diethylthiocarbamoylthioacetate exists as a tetrahedral compound (Ng & Kumar Das, 1997), in agreement with the spectroscopic assignment.

The title *N*-methyl-*N*-phenylthiocarbamoylthioacetate polymer, (II), crystallizes with four independent molecules; the bond dimensions involving the Sn atom are similar in the four molecules. The chain propagates on the *b*-*c* face of the unit cell, and its repeat distance

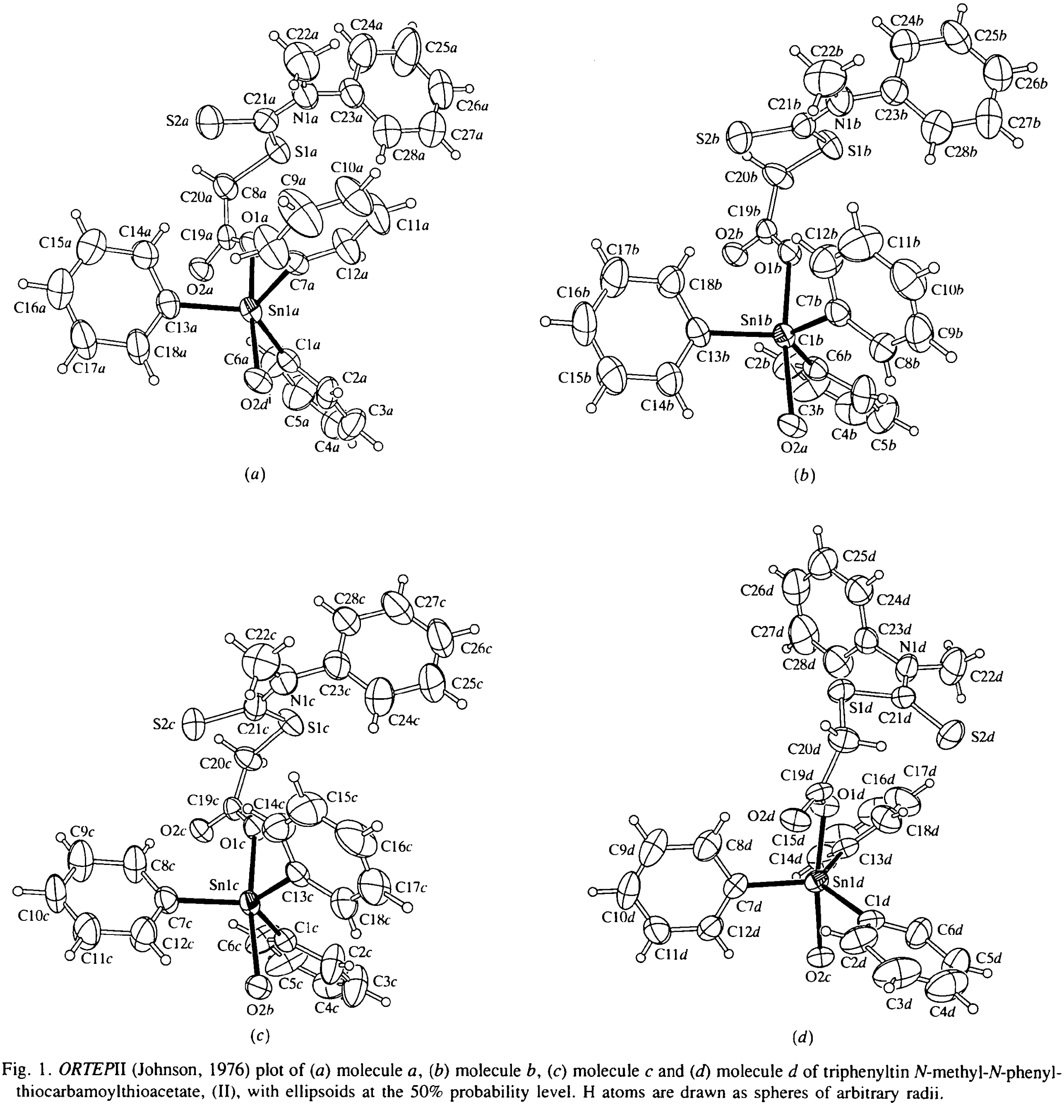
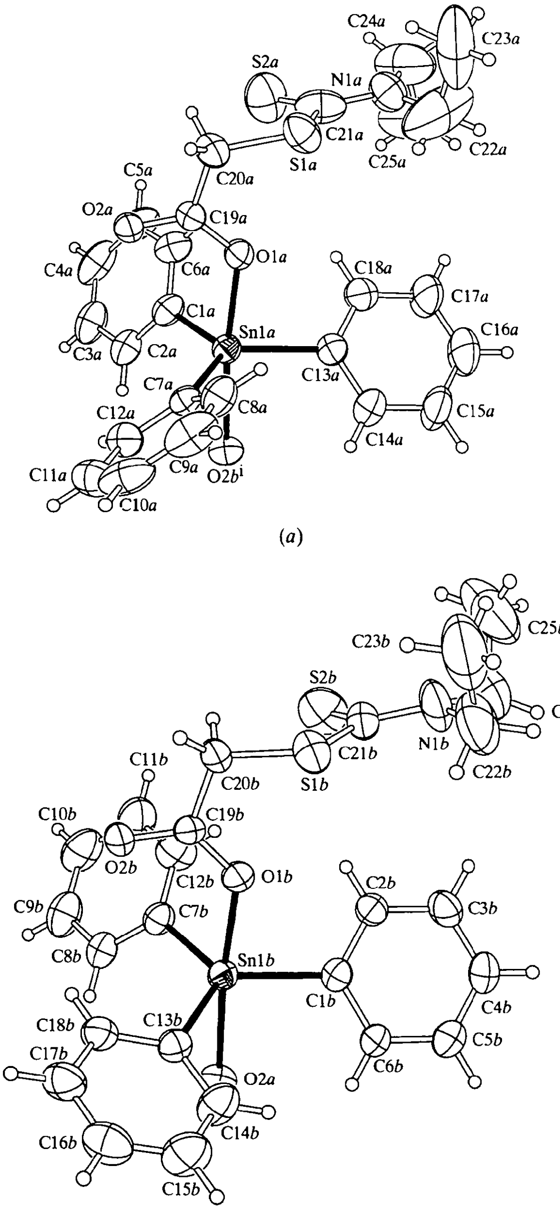


Fig. 1. *ORTEP*II (Johnson, 1976) plot of (*a*) molecule *a*, (*b*) molecule *b*, (*c*) molecule *c* and (*d*) molecule *d* of triphenyltin *N*-methyl-*N*-phenylthiocarbamoylthioacetate, (II), with ellipsoids at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

is 22.04 = 5.5 Å. The molecules are packed in such a manner that voids exist (Spek, 1990), so that the packing can be regarded as being more compact compared with that of the *N*,*N*-diethylthiocarbamoylthioacetate.

Triphenyltin *N*,*N*-tetramethylenethiocarbamoylthioacetate, (III), exists as a monomeric tetrahedral molecule.



(*a*)

(*b*)

Fig. 2. *ORTEP*II (Johnson, 1976) plot of (*a*) molecule *a* and (*b*) molecule *b* of triphenyltin *N*,*N*-diethylthiocarbamoylthioacetate, (I), with ellipsoids at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

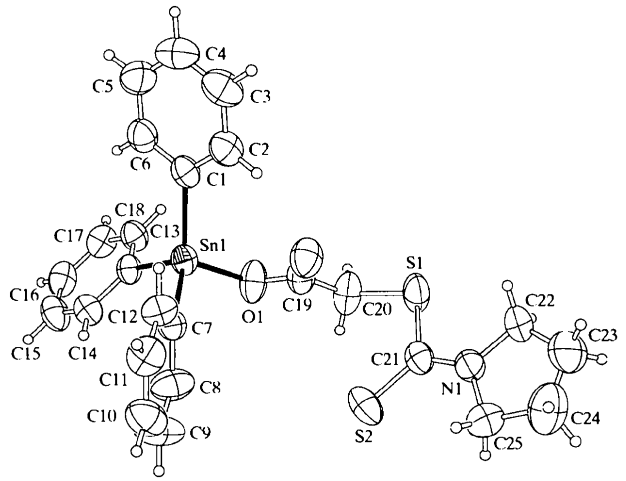


Fig. 3. *ORTEP*II (Johnson, 1976) plot of triphenyltin *N*,*N*-tetramethylenethiocarbamoylthioacetate, (III), with ellipsoids at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

Experimental

The thiocarbamoylthioacetic acids were synthesized from chloroacetic acid, carbon disulfide and the secondary amine (Nachmias, 1952), and were recrystallized from ethanol. Equimolar quantities of diethylthiocarbamoylthioacetic acid and triphenyltin hydroxide were heated in a small volume of ethanol; the crystalline organotin carboxylate (I) separated on cooling the filtered solution. The analogous reaction with *N*-methyl-*N*-phenylthiocarbamoylacetic acid and triphenyltin hydroxide, and with *N*,*N*-tetramethylenethiocarbamoylthioacetic acid yielded (II) and (III), respectively. The solid-state ^{119}Sn NMR spectrum of (II) shows a cluster of three sites at −257/−266/−278 p.p.m. (relative to tetramethyltin). The spectrum of (III) also shows a cluster of sites at −218 to −297 p.p.m.

Compound (I)

Crystal data

[$Sn(C_6H_5)_3(C_7H_{12}NO_2S_2)$]
M_r = 556.29
Monoclinic
$P2_1/c$
a = 12.900 (2) Å
b = 18.135 (2) Å
c = 21.822 (4) Å
β = 97.42 (1)°
V = 5062 (1) Å^3
Z = 8
D_x = 1.460 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 12.0–13.0°
μ = 1.195 mm^{-1}
T = 298 (2) K
Block
0.55 × 0.50 × 0.45 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.493, T_{max} = 0.584

5739 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 25°
$h = -15 \rightarrow 15$
$k = 0 \rightarrow 21$
$l = 0 \rightarrow 25$

9167 measured reflections
8911 independent reflections

3 standard reflections
frequency: 60 min
intensity decay: 4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.166$
$S = 1.009$
8911 reflections
487 parameters
H-atom parameters constrained, with $U(\mathrm{H}) = 1.5U_{\mathrm{eq}}(\mathrm{C})$

$w = 1/[\sigma^2(F_o^2) + (0.0818P)^2 + 7.1495P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 1.198$ e Å^{-3}
$\Delta\rho_{\min} = -0.598$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

Sn1*a*—C1*a*	2.125 (4)	Sn1*b*—C1*b*	2.134 (3)
Sn1*a*—C7*a*	2.113 (4)	Sn1*b*—C7*b*	2.115 (4)
Sn1*a*—C13*a*	2.144 (4)	Sn1*b*—C13*b*	2.115 (4)
Sn1*a*—O1*a*	2.186 (5)	Sn1*b*—O1*b*	2.206 (5)
Sn1*a*—O2*b*$^{\mathrm{i}}$	2.326 (5)	Sn1*b*—O2*a*	2.343 (5)
C1*a*—Sn1*a*—C7*a*	122.8 (2)	C1*b*—Sn1*b*—C7*b*	119.0 (2)
C1*a*—Sn1*a*—C13*a*	118.3 (2)	C1*b*—Sn1*b*—C13*b*	119.9 (2)
C1*a*—Sn1*a*—O1*a*	95.4 (2)	C1*b*—Sn1*b*—O1*b*	86.4 (2)
C1*a*—Sn1*a*—O2*b*$^{\mathrm{i}}$	91.8 (2)	C1*b*—Sn1*b*—O2*a*	88.8 (2)
C7*a*—Sn1*a*—C13*a*	118.5 (2)	C7*b*—Sn1*b*—C13*b*	120.8 (2)
C7*a*—Sn1*a*—O1*a*	93.2 (2)	C7*b*—Sn1*b*—O1*b*	92.9 (2)
C7*a*—Sn1*a*—O2*b*$^{\mathrm{i}}$	84.8 (2)	C7*b*—Sn1*b*—O2*a*	92.1 (2)
C13*a*—Sn1*a*—O1*a*	86.9 (2)	C13*b*—Sn1*b*—O1*b*	95.9 (2)
C13*a*—Sn1*a*—O2*b*$^{\mathrm{i}}$	87.7 (2)	C13*b*—Sn1*b*—O2*a*	83.9 (2)
O1*a*—Sn1*a*—O2*b*$^{\mathrm{i}}$	172.4 (2)	O1*b*—Sn1*b*—O2*a*	174.4 (2)
C19*a*—O1*a*—Sn1*a*	126.2 (5)	C19*b*—O1*b*—Sn1*b*	129.3 (5)
C19*a*—O2*a*—Sn1*b*	140.6 (5)	C19*b*—O2*b*—Sn1*a*$^{\mathrm{ii}}$	131.3 (5)

Symmetry codes: (i) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$; (ii) $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$.

Compound (II)

Crystal data

$[\mathrm{Sn}(\mathrm{C_6H_5})_3(\mathrm{C_{10}H_{10}NO_2S_2})]$
$M_r = 590.30$
Triclinic
$P\bar{1}$
$a = 17.019$ (2) Å
$b = 18.466$ (2) Å
$c = 18.921$ (2) Å
$\alpha = 71.952$ (7)°
$\beta = 72.482$ (8)°
$\gamma = 73.333$ (9)°
$V = 5267.1$ (9) Å^3
$Z = 8$
$D_x = 1.489$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 12.25$–$12.75°$
$\mu = 1.154$ mm^{-1}
$T = 298$ (2) K
Plate
0.44 × 0.44 × 0.11 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.512$, $T_{\max} = 0.881$
19 187 measured reflections
18 499 independent reflections

11 846 reflections with $I > 2\sigma(I)$
$R_{\mathrm{int}} = 0.026$
$\theta_{\max} = 25°$
$h = 0 \rightarrow 20$
$k = -21 \rightarrow 21$
$l = -21 \rightarrow 22$
3 standard reflections
frequency: 60 min
intensity decay: 3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.124$
$S = 1.002$
18 499 reflections
931 parameters
H-atom parameters constrained, with $U(\mathrm{H}) = 1.5U_{\mathrm{eq}}(\mathrm{C})$

$w = 1/[\sigma^2(F_o^2) + (0.0538P)^2 + 1.1698P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.759$ e Å^{-3}
$\Delta\rho_{\min} = -0.775$ 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*	2.145 (3)	Sn1*c*—C1*c*	2.131 (3)
Sn1*a*—C7*a*	2.125 (3)	Sn1*c*—C7*c*	2.159 (3)
Sn1*a*—C13*a*	2.161 (3)	Sn1*c*—C13*c*	2.145 (3)
Sn1*a*—O1*a*	2.187 (4)	Sn1*c*—O1*c*	2.190 (2)
Sn1*a*—O2*d*$^{\mathrm{i}}$	2.359 (2)	Sn1*c*—O2*b*	2.354 (2)
Sn1*b*—C1*b*	2.136 (3)	Sn1*d*—C1*d*	2.130 (3)
Sn1*b*—C7*b*	2.143 (3)	Sn1*d*—C7*d*	2.151 (3)
Sn1*b*—C13*b*	2.169 (3)	Sn1*d*—C13*d*	2.123 (3)
Sn1*b*—O1*b*	2.197 (2)	Sn1*d*—O1*d*	2.188 (2)
Sn1*b*—O2*a*	2.317 (4)	Sn1*d*—O2*c*	2.396 (2)
C1*a*—Sn1*a*—C7*a*	120.0 (2)	C1*c*—Sn1*c*—C7*c*	128.1 (2)
C1*a*—Sn1*a*—C13*a*	129.3 (1)	C1*c*—Sn1*c*—C13*c*	119.9 (1)
C1*a*—Sn1*a*—O1*a*	95.2 (2)	C1*c*—Sn1*c*—O1*c*	91.7 (1)
C1*a*—Sn1*a*—O2*d*$^{\mathrm{i}}$	86.6 (2)	C1*c*—Sn1*c*—O2*b*	86.4 (1)
C7*a*—Sn1*a*—C13*a*	109.8 (1)	C7*c*—Sn1*c*—C13*c*	111.0 (1)
C7*a*—Sn1*a*—O1*a*	87.0 (2)	C7*c*—Sn1*c*—O1*c*	97.8 (1)
C7*a*—Sn1*a*—O2*d*$^{\mathrm{i}}$	85.7 (2)	C7*c*—Sn1*c*—O2*b*	90.4 (1)
C13*a*—Sn1*a*—O1*a*	96.0 (1)	C13*c*—Sn1*c*—O1*c*	90.5 (1)
C13*a*—Sn1*a*—O2*d*$^{\mathrm{i}}$	88.7 (2)	C13*c*—Sn1*c*—O2*b*	82.7 (1)
O1*a*—Sn1*a*—O2*d*$^{\mathrm{i}}$	172.3 (2)	C19*c*—O1*c*—Sn1*c*	135.0 (2)
C19*a*—O1*a*—Sn1*a*	129.0 (3)	C19*b*—O2*b*—Sn1*c*	137.2 (1)
C19*d*—O2*d*—Sn1*a*$^{\mathrm{ii}}$	141.3 (1)	C1*d*—Sn1*d*—C13*d*	116.2 (1)
C1*b*—Sn1*b*—C7*b*	119.8 (2)	C1*d*—Sn1*d*—C7*d*	125.7 (2)
C1*b*—Sn1*b*—C13*b*	126.5 (1)	C1*d*—Sn1*d*—O1*d*	94.8 (1)
C1*b*—Sn1*b*—O1*b*	94.2 (1)	C1*d*—Sn1*d*—O2*c*	84.3 (1)
C1*b*—Sn1*b*—O2*a*	84.8 (2)	C7*d*—Sn1*d*—C13*d*	117.2 (1)
C7*b*—Sn1*b*—C13*b*	112.8 (1)	C7*d*—Sn1*d*—O1*d*	97.4 (1)
C7*b*—Sn1*b*—O1*b*	87.8 (1)	C7*d*—Sn1*d*—O2*c*	91.3 (1)
C7*b*—Sn1*b*—O2*a*	84.9 (2)	C13*d*—Sn1*d*—O1*d*	86.9 (1)
C13*b*—Sn1*b*—O1*b*	96.7 (1)	C13*d*—Sn1*d*—O2*c*	84.6 (1)
C13*b*—Sn1*b*—O2*a*	91.1 (1)	O1*d*—Sn1*d*—O2*c*	169.9 (1)
O1*b*—Sn1*b*—O2*a*	171.0 (1)	C19*d*—O1*d*—Sn1*d*	128.0 (1)
C19*b*—O1*b*—Sn1*b*	130.5 (1)	C19*c*—O2*c*—Sn1*d*	140.6 (2)
C19*a*—O2*a*—Sn1*b*	140.3 (3)		

Symmetry codes: (i) $x, y - 1, 1 + z$; (ii) $x, 1 + y, z - 1$.

Compound (III)

Crystal data

$[\mathrm{Sn}(\mathrm{C_6H_5})_3(\mathrm{C_7H_{10}NO_2S_2})]$
$M_r = 554.27$
Triclinic
$P\bar{1}$
$a = 9.7442$ (3) Å
$b = 9.8923$ (5) Å
$c = 13.3187$ (4) Å
$\alpha = 107.064$ (4)°
$\beta = 95.246$ (3)°
$\gamma = 97.153$ (4)°
$V = 1206.62$ (8) Å^3
$Z = 2$
$D_x = 1.526$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 14.0$–$16.0°$
$\mu = 1.253$ mm^{-1}
$T = 298$ (2) K
Block
0.43 × 0.22 × 0.22 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.677$, $T_{\max} = 0.738$
4532 measured reflections
4259 independent reflections
3971 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.009$
$\theta_{\max} = 25.06°$
$h = 0 \rightarrow 11$
$k = -11 \rightarrow 11$
$l = -15 \rightarrow 15$
3 standard reflections
frequency: 60 min
intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.021$
$wR(F^2) = 0.057$
$S = 1.003$
4259 reflections
280 parameters
H-atom parameters constrained, with $U(\rm H) = 1.5U_{\rm eq}(\rm C)$
$w = 1/[\sigma^2(F_o^2) + (0.0340P)^2 + 0.4226P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.320$ e Å^{-3}
$\Delta\rho_{\min} = -0.281$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

Sn1—O1	2.055 (2)	Sn1—C7	2.118 (2)
Sn1—C1	2.117 (2)	Sn1—C13	2.125 (2)
C1—Sn1—C7	115.2 (1)	C7—Sn1—C13	113.2 (1)
C1—Sn1—C13	111.0 (1)	C7—Sn1—O1	107.4 (1)
C1—Sn1—O1	113.7 (1)	C13—Sn1—O1	94.6 (1)

Phenyl rings were refined as rigid hexagons in (I) and (II). In (I), the group was restrained with C—C = 1.54±0.01, N—C = 1.45±0.01, N···C = 2.44±0.02 and C···C = 2.51±0.02 Å; a *FLAT* instruction was imposed on the S_2CNC_2 unit. The final difference map had a peak of 1 e Å^{-3} around the dithiocarbamoyl group of molecule *a*.

For all compounds, data collection: *CAD-4/PC* (Kretschmar, 1994); cell refinement: *CELDIM* in *CAD-4 VAX/PC* (Enraf–Nonius, 1988); data reduction: *XCAD4* (Harms, 1997); program(s) used to solve structures: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structures: *SHELXL97* (Sheldrick, 1997*b*); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1233). Services for accessing these data are described at the back of the journal.

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