

Data collection: *MSCIAFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSCIAFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1994). Program(s) used to solve structure: *SAPI91* (Fan, 1991). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *ORTEPII* (Johnson, 1976) in *TEXSAN*. 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: FG1551). Services for accessing these data are described at the back of the journal.

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(*N*-Maleoylmethioninato)trimethyltin(IV)†

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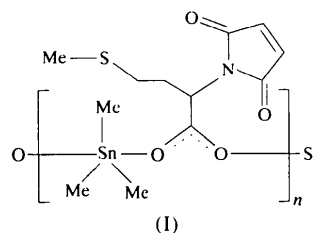
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Abstract

The crystal structure of $[\text{Sn}(\text{CH}_3)_3(\text{C}_9\text{H}_{10}\text{NO}_4\text{S})]_n$ contains polymeric chains wherein each trimethyltin moiety bridges two neighboring *N*-maleoylmethioninate ligands via carboxyl moieties. The Sn atom has a distorted trigonal-bipyramidal geometry, with three methyl groups in the equatorial plane [mean Sn—C 2.126 (6) Å]. The carboxyl O atoms bonded to the Sn atom in the axial positions have significantly different Sn—O bond lengths [2.152 (6) and 2.484 (6) Å].

Comment

The structural chemistry of organotin compounds with a coordination number higher than four is being extensively studied because of their biological activity, enhanced reactivity and stereochemical non-rigidity (Mehring *et al.*, 1998). Several reports have been cited in the literature regarding the structural chemistry of triorganotin carboxylate complexes in which the carboxylates are amino acids or protected amino acids (Tiekink, 1994). As a continuation of our work on the structural chemistry of organotin carboxylates (Parvez *et al.*, 1997; Tahir, Ülkü, Ali *et al.*, 1997; Tahir, Ülkü, Danish *et al.*, 1997; Bhatti *et al.*, 1999), we now report the crystal structure of (*N*-maleoylmethioninato)trimethyltin, (I).



The structure of (I) is presented in Fig. 1. The Sn atom has three methyl groups bonded in the equatorial plane. The Sn—C distances, which are almost identical, lie in the range 2.121 (9)–2.130 (9) Å and are in

† Alternative name: *catena*-poly[[trimethyltin(IV)]- μ -[2-(2,5-dioxo-3-pyrazolin-1-yl)-4-methylthiobutanoato-*O*:*O'*]].

agreement with the values reported for related structures. The Sn—O bonds in the axial positions involving one carboxyl O atom [Sn1—O1 2.152(6) Å] and another symmetry-related carbonyl O atom in a *trans* position [Sn1—O2 2.484(6) Å] are significantly different from each other; the O—Sn—O angle is 174.4(2)°.

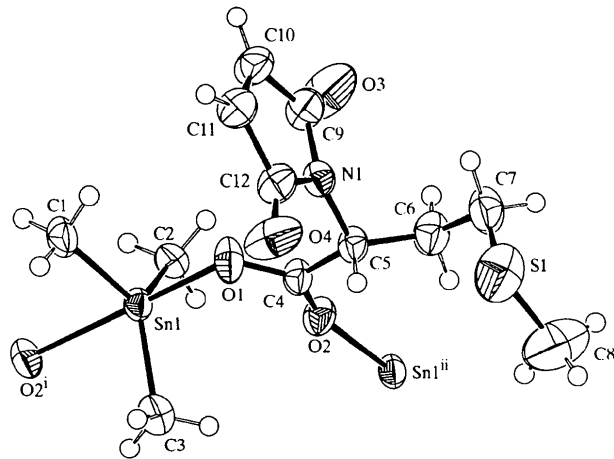


Fig. 1. ORTEP (Johnson, 1976) drawing of (I) with the atomic numbering scheme. Displacement ellipsoids have been plotted at the 50% probability level and H atoms have been assigned arbitrary radii. [Symmetry codes: (i) $\frac{3}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (ii) $\frac{3}{2} - x, y + \frac{1}{2}, \frac{1}{2} - z$.]

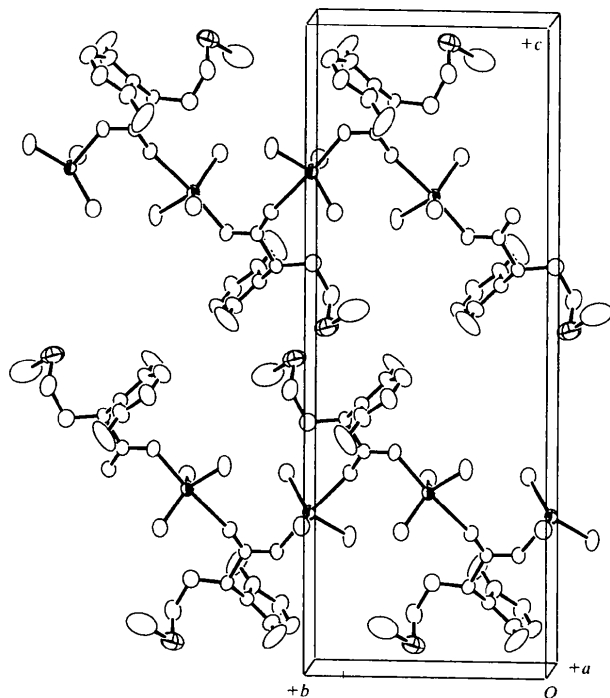


Fig. 2. Unit-cell packing of (I) showing polymeric chains running along the *b* axis. H atoms have been omitted for clarity.

The Sn atom has a distorted trigonal-bipyramidal geometry, with the Sn atom 0.128(6) Å out of the equatorial plane formed by the three methyl-group C atoms towards the more strongly bound O1 atom. The structure is composed of polymeric chains running along the *b* axis (Fig. 2). The molecular dimensions in the *N*-maleoylmethioninate ligand are normal.

The crystal structures of tin complexes of some related compounds which have been reported in the literature include: (*N*-phthaloyl-leucinato)triphenyltin, wherein tin shows a tetrahedral geometry (Ng *et al.*, 1990), (glycinato)trimethyltin, in which tin adopts a polymeric distorted trigonal-bipyramidal geometry with O and N donor atoms (Ho *et al.*, 1980), and (glutamato)trimethyltin, with tin exhibiting a polymeric distorted trigonal-bipyramidal geometry as a result of a bridging carboxylate group (Huber *et al.*, 1989). Recently, a few structures having the general formula [R₃SnOCOR'(H₂O)] have been reported (Ng *et al.*, 1994; Lo *et al.*, 1997). Another interesting structure, (*N*-phthaloylglycinato)tricyclohexanetin, shows a tetrahedral geometry which is distorted towards a trigonal bipyramid (Ng & Kumar Das, 1997).

Experimental

The title compound was prepared by reacting equimolar amounts of the triethylammonium salt of *N*-maleoylmethionine and trimethyltin chloride in toluene under reflux conditions for 4 h (Bhatti *et al.*, 1999). Triethylamine hydrochloride was removed by filtration and the solid obtained after removal of the solvent was crystallized from dichloromethane.

Crystal data

[Sn(CH₃)₃(C₉H₁₀NO₄S)]
M_r = 392.03
 Monoclinic
*P*2₁/*n*
a = 6.507(5) Å
b = 9.640(3) Å
c = 26.376(3) Å
 β = 91.51(3)°
V = 1653.9(14) Å³
Z = 4
D_x = 1.574 Mg m⁻³
D_m not measured

Mo *K*α radiation
 λ = 0.71069 Å
 Cell parameters from 25 reflections
 θ = 10–15°
 μ = 1.68 mm⁻¹
T = 170(1) K
 Prism
 0.55 × 0.51 × 0.22 mm
 Colorless

Data collection

Rigaku AFC-6S diffractometer
 ω -2 θ scans
 Absorption correction:
 empirical ψ scan (3 reflections; North *et al.*, 1968)
 T_{\min} = 0.426, T_{\max} = 0.691
 4045 measured reflections
 3776 independent reflections

2849 reflections with $I > 2\sigma(I)$
 R_{int} = 0.027
 θ_{max} = 27.57°
 $h = 0 \rightarrow 8$
 $k = 0 \rightarrow 12$
 $l = -34 \rightarrow 34$
 3 standard reflections every 200 reflections
 intensity decay: <2%

Refinement

Refinement on F^2	$(\Delta/\sigma)_{\max} < 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.059$	$\Delta\rho_{\max} = 1.43 \text{ e } \text{\AA}^{-3}$
$wR(F^2) = 0.159$	$\Delta\rho_{\min} = -1.00 \text{ e } \text{\AA}^{-3}$
$S = 1.224$	Extinction correction:
3776 reflections	<i>SHELXL97</i>
173 parameters	Extinction coefficient:
H atoms fixed with an	0.0002 (2)
overall U_{iso}	Scattering factors from
$w = 1/[\sigma^2(F_o^2) + 19.82P]$	<i>International Tables for</i>
where $P = (F_o^2 + 2F_c^2)/3$	<i>Crystallography</i> (Vol. C)

Table 1. Selected geometric parameters (\AA , $^\circ$)

Sn1—C1	2.121 (9)	Sn1—O2'	2.484 (6)
Sn1—C3	2.127 (10)	S1—C7	1.771 (13)
Sn1—C2	2.130 (9)	S1—C8	1.800 (15)
Sn1—O1	2.152 (6)		
C1—Sn1—C3	117.0 (4)	C3—Sn1—O2'	83.9 (3)
C1—Sn1—C2	115.5 (4)	C2—Sn1—O2'	87.9 (3)
C3—Sn1—C2	126.5 (4)	O1—Sn1—O2'	174.4 (2)
C1—Sn1—O1	88.6 (3)	C7—S1—C8	101.1 (7)
C3—Sn1—O1	93.7 (3)	C4—O1—Sn1	122.6 (5)
C2—Sn1—O1	97.6 (3)	C4—O2—Sn1 ^{II}	164.6 (6)
C1—Sn1—O2'	88.0 (3)		

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

The space group $P2_1/n$ was determined uniquely from the systematic absences. The final difference map had a peak of $1.43 \text{ e } \text{\AA}^{-3}$ close to the Sn atom (1.4 \AA) and was of no chemical significance.

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Dimeric (pyrazole- N^2)(N -salicylidene-2,2-dimethylglycinato- O, N, O')copper(II) pyrazole solvate

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Abstract

In the title compound, bis(μ - N -salicylidene-2,2-dimethylglycinato)bis[(pyrazole)copper(II)] bis(pyrazole) solvate, $[\text{Cu}_2(\text{C}_{11}\text{H}_{11}\text{NO}_3)_2(\text{C}_3\text{H}_4\text{N}_2)_2] \cdot 2\text{C}_3\text{H}_4\text{N}_2$, the Cu^{II} atom adopts a square-pyramidal copper(II) coordination with both a tridentate 2,2-dimethyl- N -salicylidene-glycinato Schiff base dianion and a pyrazole ligand forming the basal plane. The apex of the pyramid is occupied by an O atom from an adjacent chelate molecule at a distance of 2.605 (2) \AA , building a centrosymmetric dimer. All copper polyhedra belonging to one dimer are oriented parallel to each other, whereas the dimers are ordered antiferrodistortively.