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Tri-*n*-butyl(*N*-maleoyl-β-alaninato)tin†

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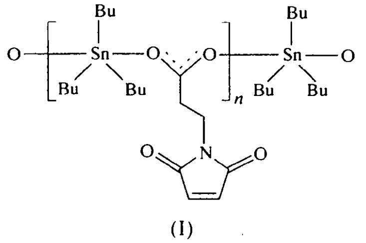
Abstract

The Sn atom in $[Sn(C_4H_9)_3(C_7H_6NO_4)]_n$ adopts a distorted trigonal-bipyramidal geometry, with three *n*-butyl groups defining the trigonal plane [mean Sn—C distance 2.145 (1) Å] and the axial positions occupied by the O atoms of different carboxylate groups having significantly different Sn—O bond lengths [2.215 (5) and 2.424 (5) Å]. The structure forms a polymeric chain of complex molecules linked *via* carboxylate moieties.

Comment

The structural chemistry of triorganotin complexes of amino acids and protected amino acids has revealed a wide variety of features and a rich diversity of structural motifs. For example, in the crystal structures of aquatributyltin (*N*-phthaloylglycinate) (Ho *et al.*, 1980), tricyclohexyl(*N*-phthaloylglycinato)tin (Ng & Kumar Das, 1997*a*), tricyclohexyl[(*N*,*N*-diethylthiocarbamoylthio)-acetato-*O*]tin (Ng & Kumar Das, 1997*b*) and aquatrimethyltin 4-(4-chlorophenyl)-2-phenyl-5-thiazoleacetate (Parvez, Ali, Mazhar, Bhatti & Khokhar, 1999), Sn atoms show distorted bipyramidal geometry. On the other hand, the Sn atom adopts a tetrahedral geometry in the structure of *N*-phthaloylleucinate triphenyltin (Ng *et al.*, 1990). In a continuation of our work on the structural chemistry of organotin complexes of carboxylate-protected amino acids (Bhatti *et al.*, 1999; Parvez, Ali, Mazhar, Bhatti & Khokhar, 1999; Parvez, Ali, Mazhar, Bhatti & Choudhary, 1999), we now report the crystal structure of tri-*n*-butyl(*N*-maleoyl-β-alaninato)tin, (I).



(I)

The structure of (I) is presented in Fig. 1. The Sn atom has three *n*-butyl groups bonded in equatorial positions, with Sn—C distances [mean 2.145 (8) Å] equal within experimental error and in agreement with the values reported for related structures (Allen *et al.*, 1983). The axial positions are occupied by O atoms of carboxylate residues, with a linear O—Sn—O angle of 171.88 (17)°. The intramolecular Sn1—O1 separation of 2.215 (5) Å is significantly shorter than the intermolecular Sn1—O2^{i} distance of 2.424 (5) Å [symmetry code: (i) $\frac{1}{2}+x, \frac{1}{2}-y, z$], indicating that the former is a covalent bond and the latter is a coordinate bond. The Sn atom has a distorted trigonal-bipyramidal geometry, with the Sn atom 0.149 (5) Å out of the equatorial plane formed by the three methyl C atoms towards the more strongly bound O1 atom.

The structure is composed of infinite chains of (I) running parallel to the *a* axis. The molecular dimensions in the *N*-maleoyl-β-alaninate ligand are normal.

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

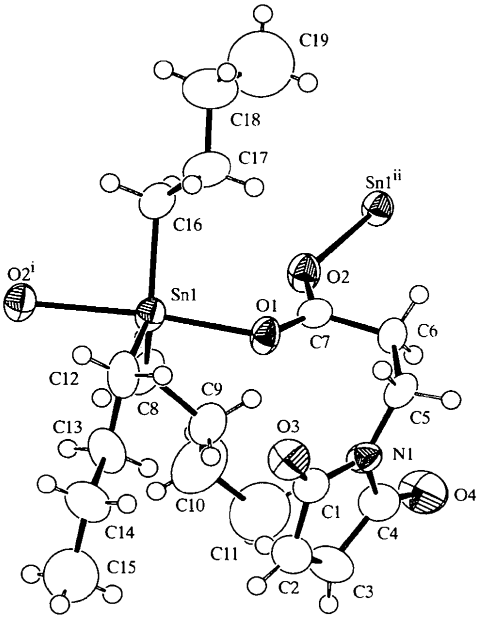

Fig. 1. *ORTEP*II (Johnson, 1976) drawing of (I) with the atomic numbering scheme. Displacement ellipsoids have been plotted at the 50% probability level and H atoms have been assigned arbitrary radii. The minor fractions of the disordered *n*-butyl groups have been ignored. [Symmetry codes: (i) $\frac{1}{2}+x$,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

A few examples of crystal structures containing triorganotin complexes of protected compounds forming polymeric chains similar to (I) are: (3-indolylacetato-*O*,*O*′)tri-*n*-butyltin (Molloy *et al.*, 1987), (glutamato-*O*,*O*′)trimethyltin (Huber *et al.*, 1989), [3-(2-thienyl)-2-propenoato]triethyltin (Danish *et al.*, 1995), 2-[(2,3-dimethylphenyl)aminobenzoato-*O*,*O*′]trimethyltin (Tahir *et al.*, 1997), (picolinato *N*-oxide)triphenyltin and (nicotinato *N*-oxide)triphenyltin (Ng & Kumar Das, 1995), triphenyl(3-ureidopropionato-*O*,*O*′)tin (Lo *et al.*, 1991) and (*N*-salicylidene-6-aminohexanoato-*O*,*O*′)triphenyltin (Toong *et al.*, 1992).

Experimental

The *N*-maleoyl-β-alaninate ligand was prepared by stirring equimolar quantities of maleic anhydride and β-alanine in acetic acid for 3 h (Rich *et al.* 1975). The solid maleamic acid was filtered off, washed with cold water and air dried. Maleamic acid (16.8 mmol) was suspended in dry toluene and treated with triethylamine (35.5 mmol). The mixture was refluxed with vigorous stirring for 4 h. The water which formed was continuously removed *via* a Dean–Stark apparatus. After cooling, toluene was removed from the orange laer, tributyltin chloride (16.8 mmol) was added in toluene and the mixture was refluxed for a further 3–4 h. On cooling, triethylammonium chloride was filtered off, the toluene solvent removed by rotary evaporation and the residue was recrystallized from dichloromethane solution giving thin needles of the title compound.

Crystal data

$[Sn(C_4H_9)_3(C_7H_6NO_4)]$
$M_r = 458.15$
Monoclinic
$P2_1/a$
$a = 10.574\,(3)$ Å
$b = 16.812\,(2)$ Å
$c = 12.882\,(2)$ Å
$\beta = 102.15\,(2)°$
$V = 2238.6\,(8)$ Å^3
$Z = 4$
$D_x = 1.359$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 6.5–15.0°$
$\mu = 1.161$ mm^{-1}
$T = 170\,(1)$ K
Needle
0.47 × 0.11 × 0.10 mm
Colorless

Data collection

Rigaku AFC-6*S* diffractometer
ω–2θ scans
Absorption correction: empirical ψ scan (3 reflections; North *et al.*, 1968)
$T_{min} = 0.611$, $T_{max} = 0.893$
4199 measured reflections
3969 independent reflections

2363 reflections with $I > 2\sigma(I)$
$R_{int} = 0.088$
$\theta_{max} = 25.05°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 20$
$l = -15 \rightarrow 15$
3 standard reflections every 200 reflections intensity decay: 4.20%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.141$
$S = 1.020$
3969 reflections
230 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.057P)^2 + 7.52P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.683$ e Å^{-3}
$\Delta\rho_{min} = -0.826$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

Sn1—C8	2.145 (8)	O2—C7	1.263 (8)
Sn1—C12	2.146 (8)	O3—C1	1.190 (8)
Sn1—C16	2.144 (7)	O4—C4	1.197 (10)
Sn1—O1	2.215 (5)	N1—C4	1.387 (9)
Sn1—O2^{i}	2.424 (5)	N1—C1	1.404 (9)
O1—C7	1.262 (8)	N1—C5	1.457 (9)
C16—Sn1—C8	121.8 (4)	C12—Sn1—O2^{i}	83.8 (2)
C16—Sn1—C12	121.4 (3)	O1—Sn1—O2^{i}	171.88 (17)
C8—Sn1—C12	115.3 (4)	C7—O1—Sn1	121.7 (5)
C16—Sn1—O1	96.5 (2)	C7—O2—Sn1^{ii}	136.7 (4)
C8—Sn1—O1	97.1 (3)	C4—N1—C1	110.7 (6)
C12—Sn1—O1	88.1 (3)	C4—N1—C5	124.4 (6)
C16—Sn1—O2^{i}	88.3 (2)	C1—N1—C5	124.9 (6)
C8—Sn1—O2^{i}	85.8 (3)		

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

Two of the *n*-butyl groups were disordered with the C13–C15 and C19 atoms located over two sites each with unequal site-occupancy factors [0.660 (11)/0.340 (11) and 0.644 (18)/0.356 (18), respectively]; the minor sites have been primed in the atomic labels.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1994). Program(s) used to solve structure: *SAPI*91 (Fan, 1991). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ORTEP*II (Johnson, 1976) in *TEXSAN*. Software used to prepare material for publication: *SHELXL*97.

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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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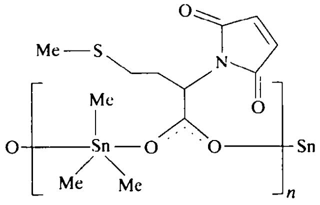
Abstract

The crystal structure of $[Sn(CH_3)_3(C_9H_{10}NO_4S)]_n$ 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).



(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

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