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catena-Poly[[tri-*n*-butyltin]-μ-*N*-(1-naphthyl)maleamato]

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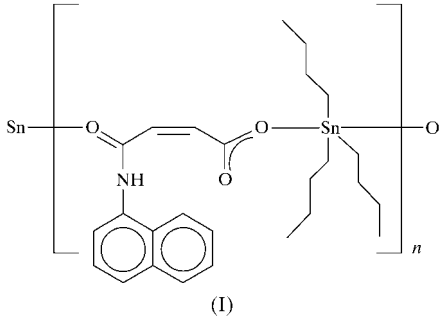
The crystal structure of *catena*-poly[[tri-*n*-butyltin]-μ-3-(1-naphthylaminocarbonyl)acrylato-$\kappa^2O^1{:}O^3$], $[Sn(C_4H_9)_3(C_{14}H_{10}NO_3)]_n$, is composed of polymeric chains wherein the metal center exhibits a distorted trigonal-bipyramidal geometry, with three *n*-butyl groups defining the trigonal plane [mean Sn—C 2.133 (7) Å] and the axial positions being occupied by the carboxylate O atoms of two different *N*-(1-naphthyl)maleamate ligands with inequivalent Sn—O distances [2.167 (4) and 2.457 (4) Å]. The *N*-(1-naphthyl)-maleamate fragment forms an essentially planar seven-membered ring involving an intramolecular N—H···O hydrogen bond.

Comment

There have been several reports dealing with the impact of organotin chemistry in the biosphere (Gielen, 1994; Ng *et al.*, 1991). Exploration of the structure–activity relationships of such systems has led to numerous reports in recent years (Gielen *et al.*, 1994; Selvaratnam *et al.*, 1994; McManus *et al.*, 1994). Furthermore, the structural chemistry of organotin compounds with a coordination number greater than four is being extensively studied because of the biological activity, enhanced reactivity and stereochemical non-rigidity of these



(I)

compounds (Mehring *et al.*, 1998). Our contributions in this field have concerned the synthesis and structural characterization of organotin derivatives of donor ligands containing chalcogens, with special reference to their biological applications (Badshah *et al.*, 1994; Danish *et al.*, 1995; Ali *et al.*, 1993; Choudhary *et al.*, 2001; Bhatti *et al.*, 2000). In a continuation of our studies on the structural chemistry of organotin carboxylates (Parvez, Ali, Mazhar, Bhatti & Khokhar, 1999; Parvez, Ali, Mazhar, Bhatti & Choudhary, 1999; Parvez *et al.*, 2000), we have prepared *catena*-poly[[tri-*n*-butyltin]-μ-*N*-(1-naphthyl)maleamato], (I), the crystal structure of which is reported here.

The structure of (I) (Fig. 1) is composed of polymeric chains wherein O atoms of both ends of the *N*-(1-naphthyl)-maleamate ligand coordinate the Sn atoms of trigonal-planar tri-*n*-butyltin moieties. The Sn—C distances (Table 1) are identical within 3σ limits [mean Sn—C 2.133 (7) Å]. On the other hand, the Sn—O1 and Sn—O3 distances are significantly different from one another, with values of 2.167 (5) and 2.457 (4) Å, respectively, 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 0.153 (2) Å out of the equatorial plane formed by the three C atoms bonded to it, towards the more strongly bonded O1 atom. The O1—Sn1—O3 angle is almost linear [177.97 (17)°], the C—Sn—C angles deviate somewhat from the ideal value of 120° [range 114.5 (3)–128.5 (3)°] and the O—Sn—C angles lie in the range 82.1 (2)–97.8 (2)°. These bond distances and angles are in agreement with the corresponding values found for similar Sn complexes contained in the Cambridge Structural Database (Allen & Kennard, 1993).

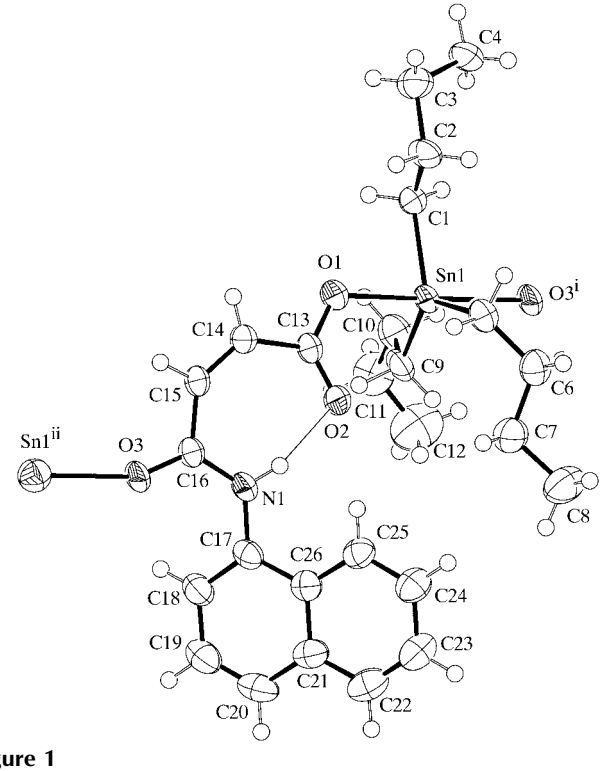


Figure 1
*ORTEP*II (Johnson, 1976) drawing of (I) with displacement ellipsoids plotted at the 50% probability level. [Symmetry codes: (i) $x+1, y, z$; (ii) $x-1, y, z$.]

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The molecular dimensions in the *N*-(1-naphthyl)maleamate ligand are normal. The ligand forms an essentially planar seven-membered ring involving an intramolecular N—H···O hydrogen bond [H1···O2 1.82 Å, N1···O1 2.669 (7) Å and N—H···O 162°]. In terms of graph-set representation (Bernstein *et al.*, 1994), this ring exhibits an *S*(7) hydrogen-bond pattern (Table 2).

Experimental

N-(1-Naphthyl)maleamic acid was prepared by adding 1-naphthylamine (8.59 g, 0.06 mol) to a solution containing an equimolar quantity of maleic anhydride (5.88 g, 0.06 mol) in glacial acetic acid (100 ml). The reaction mixture was stirred for 1 h, which afforded a yellow solid. The yellow product was washed with water, dried and recrystallized from ethanol–water (m.p. 421–422 K). The silver salt of *N*-(1-naphthyl)maleamic acid was prepared by dissolving the acid (4.82 g, 0.02 mol) in ethanol (200 ml) and adding to it a solution of an equimolar quantity of sodium bicarbonate (1.68 g, 0.02 mol). A solution of silver nitrate (3.397 g, 0.02 mol) was added dropwise to the above mixture. The precipitates which formed were filtered off under suction, washed with water and dried over anhydrous calcium chloride in the dark. The silver salt (1.735 g, 0.005 mol) was suspended in dry chloroform (50 ml) in a 250 ml two-neck round-bottomed flask equipped with a magnetic stirrer bar and a condenser. Tributyltin chloride (1.22 ml, 0.005 mol) was added slowly with constant stirring. The reaction mixture was refluxed for 8 h under an inert atmosphere. Silver chloride which formed during the reaction was removed by filtration and the solvent was evaporated under reduced pressure. Purified (I) was obtained by crystallization of the product from chloroform–*n*-hexane (1:1) at room temperature.

Crystal data

$[Sn(C_4H_9)_3(C_{14}H_{10}NO_3)]$	$Z = 2$
$M_r = 530.26$	$D_x = 1.377$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 9.5705$ (7) Å	Cell parameters from 8025 reflections
$b = 11.7377$ (9) Å	$\theta = 1.0$–$25.0°$
$c = 12.4726$ (11) Å	$\mu = 1.02$ mm^{-1}
$\alpha = 97.011$ (3)°	$T = 170$ (2) K
$\beta = 107.571$ (4)°	Block, yellow
$\gamma = 102.297$ (6)°	0.15 × 0.10 × 0.08 mm
$V = 1278.71$ (18) Å^3	

Data collection

Nonius KappaCCD diffractometer	3640 reflections with $I > 2\sigma(I)$
ω and φ scans	$R_{int} = 0.035$
Absorption correction: multi-scan (*SORTAV*: Blessing, 1997)	$\theta_{max} = 25.0°$
$T_{min} = 0.861$, $T_{max} = 0.922$	$h = -11 \rightarrow 11$
8025 measured reflections	$k = -13 \rightarrow 13$
4329 independent reflections	$l = -14 \rightarrow 14$
	Intensity decay: <0.1%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0842P)^2 + 3.9999P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.061$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.169$	$\Delta\rho_{max} = 1.80$ e Å^{-3}
$S = 1.13$	$\Delta\rho_{min} = -1.43$ e Å^{-3}
4329 reflections	
280 parameters	
H-atom parameters constrained	

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *HKL DENZO* (Otwinowski & Minor, 1997); data reduction: *HKL SCALEPACK* (Otwinowski & Minor, 1997); structure solution: *SAPI*91 (Fan, 1991); structure refinement: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976).

Table 1
Selected geometric parameters (Å, °).

Sn1—C1	2.129 (7)	O1—C13	1.293 (8)
Sn1—C9	2.127 (8)	O2—C13	1.226 (9)
Sn1—C5	2.142 (7)	O3—C16	1.231 (8)
Sn1—O1	2.167 (5)	N1—C16	1.339 (9)
Sn1—O3^{i}	2.457 (4)	N1—C17	1.412 (9)
C1—Sn1—C9	114.5 (3)	C9—Sn1—O3^{i}	82.1 (2)
C1—Sn1—C5	115.5 (3)	C5—Sn1—O3^{i}	85.8 (2)
C9—Sn1—C5	128.5 (3)	O1—Sn1—O3^{i}	177.97 (17)
C1—Sn1—O1	87.8 (2)	C13—O1—Sn1	118.6 (4)
C9—Sn1—O1	97.8 (2)	C16—O3—Sn1^{ii}	147.0 (5)
C5—Sn1—O1	95.8 (2)	C16—N1—C17	126.6 (6)
C1—Sn1—O3^{i}	90.3 (2)		

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

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2	0.88	1.82	2.669 (7)	162

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1647). Services for accessing these data are described at the back of the journal.

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