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catena-Poly[[trimethyltin(IV)]-μ-(2,5-dioxo-2,5-dihydro-1*H*-pyrrol-1-yl)-3-phenylpropionato-κ^2O:O']

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.004 Å
R factor = 0.023
wR factor = 0.060
Data-to-parameter ratio = 23.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The molecules of the title compound, [Sn(CH$_3$)$_3$(C$_{13}$H$_{10}$NO$_4$)], form polymeric chains involving both O atoms of the acid moiety. The geometry around the Sn atom is distorted trigonal bipyramidal, whith three methyl C atoms occupying the equatorial positions and O atoms from two symmetry-related molecules at axial positions.

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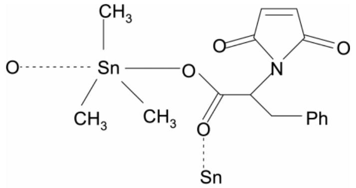
Comment

Besides the biological applications of organotin compounds, especially as antitumor and anticancer agents (Crowe, 1989; Yang & Guo, 1999; Gielen, 2002), the structural aspects of organotin compounds in general and organotin carboxylates in particular have been a main focus of active research in the past 25 years (Tiekink, 1991, 1994; Gielen, 1996; Mazhar *et al.*, 2001). Today, a number of organotin derivatives are known to be excellent anticancer agents and their structures have been well characterized in the solid state (Gielen, 1996, 2002). The developments in both biological and structural aspects of organotin derivatives of biologically active ligands with main emphasis on amino acids and peptides have been reviewed by several authors (Nath *et al.*, 2001; Chandrasekhar *et al.*, 2002; Pellerito & Nagy, 2002). Continuing our studies of the structural chemistry of biologically active organotin carboxylates (Parvez, Ali, Bhatti *et al.*, 1999; Parvez, Ali, Mazhar, Bhatti & Choudhary, 1999; Parvez, Ali, Mazhar, Bhatti & Khokhar, 1999; Parvez *et al.*, 2000, 2002), we now report the structure of the title compound, (I).



(I)

The structure of (I) consists of polymeric chains (Fig. 1) incorporating both O atoms of the acid moiety, with significantly different Sn—O distances [Sn1—O1 = 2.1517 (17) Å and Sn1—O2* = 2.5094 (17) Å; symmetry code: (*) $1 - x$, $y + \frac{1}{2}$, $\frac{3}{2} - z$], indicating that the former is a covalent bond and the latter is a coordinate bond. The geometry around the Sn atom is distorted trigonal bipyramidal, with three methyl groups occupying the positions in the equatorial plane. The Sn—C distances (Table 1) are identical within 3σ limits [mean Sn—C = 2.118 (2) Å]. Atom Sn1 lies 0.1538 (16) Å out of the equatorial plane formed by the three methyl C atoms, towards the more strongly bonded atom O1. The O—Sn—O angle is

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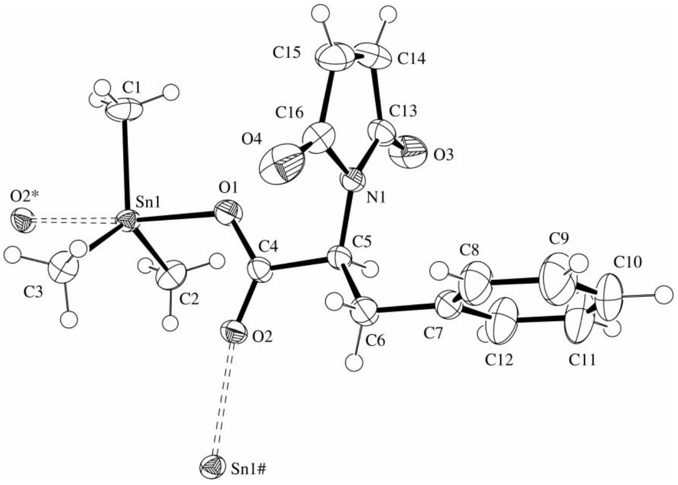

Figure 1
*ORTEP*II (Johnson, 1976) drawing of the polymeric chain of (I), with displacement ellipsoids plotted at the 50% probability level. [Symmetry codes: (*) $1 - x, y + \frac{1}{2}, \frac{3}{2} - z$; (#) $1 - x, y - \frac{1}{2}, \frac{3}{2} - z$.]

linear [173.49 (6)°], the C—Sn—C angles are between 115.86 (11) and 125.18 (11)°, deviating from the ideal value of 120°, and the O—Sn—C angles are in the range 83.21 (9)–97.56 (9)°. These dimensions are in agreement with the corresponding values reported for similar Sn complexes in the Cambridge Structural Database (Version 1.5, 2002 release; Allen, 2002). The molecular dimensions in the ligand are normal.

Experimental

The ligand was prepared by stirring equimolar quantities of maleic anhydride and L-phenylalanine [(*S*)-2-amino-3-phenylpropionic acid] in acetic acid for 3 h according to the procedure reported by Rich *et al.* (1975). Solid maleamic acid was filtered off, washed with cold water and air-dried. Maleamic acid (0.01 mol) was suspended in dry toluene and treated with triethylamine (0.025 mol). The mixture was refluxed with vigorous stirring for 4 h. The water formed was removed continuously *via* a Dean—Stark apparatus. After cooling, toluene was removed from the orange layer, trimethyltin(IV) chloride (0.01 mol) in toluene was added and the mixture was refluxed for a further 3–4 h. On cooling, triethylamine hydrochloride was filtered off and the product was recrystallized from a chloroform/*n*-hexane (1:1) mixture.

Crystal data

$[Sn(CH_3)_3(C_{13}H_{10}NO_4)]$
$M_r = 408.01$
Orthorhombic, $P2_12_12_1$
$a = 6.4920\ (10)$ Å
$b = 9.857\ (2)$ Å
$c = 28.154\ (6)$ Å
$V = 1801.6\ (6)$ Å^3
$Z = 4$
$D_x = 1.504$ Mg m^{-3}

Mo *Kα* radiation
Cell parameters from 4688 reflections
$\theta = 3.5$–$30.0°$
$\mu = 1.43$ mm^{-1}
$T = 173\ (2)$ K
Block, colorless
0.16 × 0.12 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
ω and φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
$T_{min} = 0.803$, $T_{max} = 0.894$
4688 measured reflections

4688 independent reflections
4553 reflections with $I > 2\sigma(I)$
$\theta_{max} = 30.0°$
$h = -8 \rightarrow 9$
$k = -13 \rightarrow 13$
$l = -39 \rightarrow 39$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.023$
$wR(F^2) = 0.060$
$S = 1.09$
4688 reflections
200 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + 0.58P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.41$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0084 (12)
Absolute structure: Flack (1983), 1841 Friedel pairs
Flack parameter = 0.01 (2)

Table 1
Selected geometric parameters (Å, °).

Sn1—C2	2.116 (3)	O2—Sn1^{ii}	2.5095 (17)
Sn1—C1	2.116 (3)	O3—C13	1.213 (3)
Sn1—C3	2.121 (2)	O4—C16	1.205 (3)
Sn1—O1	2.1517 (17)	N1—C13	1.385 (3)
Sn1—O2^{i}	2.5094 (17)	N1—C16	1.392 (3)
O1—C4	1.278 (3)	N1—C5	1.458 (3)
O2—C4	1.237 (3)		
C2—Sn1—C1	117.38 (12)	C3—Sn1—O2^{i}	88.73 (8)
C2—Sn1—C3	125.18 (11)	O1—Sn1—O2^{i}	173.49 (6)
C1—Sn1—C3	115.86 (11)	C4—O1—Sn1	120.51 (14)
C2—Sn1—O1	94.46 (9)	C4—O2—Sn1^{ii}	160.11 (16)
C1—Sn1—O1	90.12 (9)	C13—N1—C16	109.5 (2)
C3—Sn1—O1	97.56 (9)	C13—N1—C5	123.1 (2)
C2—Sn1—O2^{i}	83.21 (9)	C16—N1—C5	125.1 (2)
C1—Sn1—O2^{i}	85.59 (9)		

Symmetry codes: (i) $1 - x, \frac{1}{2} + y, \frac{3}{2} - z$; (ii) $1 - x, y - \frac{1}{2}, \frac{3}{2} - z$.

H atoms were located in a difference Fourier synthesis and were included in the refinement at idealized positions, with isotropic displacement parameters equal to 1.5 (methyl) or 1.2 (other H atoms) times the equivalent displacement parameters of the atoms to which they were bonded, and with C—H distances in the range 0.95–1.00 Å. The final difference map was free of any chemically significant features. The Flack (1983) parameter for the inverted structure using 1841 Friedel pairs was 0.99 (2). Therefore, the inverted structure was rejected.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *HKL DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); 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); software used to prepare material for publication: *SHELXL*97 (Sheldrick, 1997).

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