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Polymeric [2-(*N*-maleoyl)-2-methylpropionato]trimethyltin(IV)

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The structure of the title compound, $[Sn(CH_3)_3(C_8H_8NO_4)]$, is composed of two independent monomers in the asymmetric unit, which form polymeric chains involving both O atoms of the acid moieties lying parallel to the *b* axis, and are separated by one half unit cell along the *a* axis. The geometry around the Sn atoms in both monomers is distorted trigonal-bipyramidal, where three methyl C atoms occupy the equatorial positions with almost identical Sn—C distances, and O atoms from two symmetry-related ligands are in the axial positions with significantly different Sn—O distances.

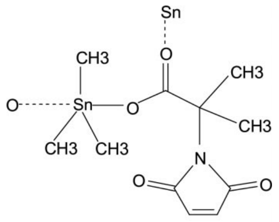
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.006 Å
R factor = 0.039
wR factor = 0.081
Data-to-parameter ratio = 20.6

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

Comment

The preparation of *N*-maleoyl amino acids and their esters has been reported (Rich *et al.*, 1975; Oishi *et al.*, 1992; Borah *et al.*, 1998). These compounds are used in the preparation of peptide–protein conjugates and a wide variety of other conjugates of biochemical interest. In recent years, polymers of *N*-substituted maleimide have gained considerable interest because of their excellent thermal, unusual chemical and oxidative stability (Rivas *et al.*, 1998). The biological applications of organotin compounds as antitumor and anticancer agents (Yang & Guo, 1999 and Gielen, 2002) and the structural aspects of organotin carboxylates, especially amino acids, their derivatives and peptides, have been well documented (Tiekink, 1991, 1994; Mazhar *et al.*, 2001; Nath *et al.*, 2001; Chandrasekhar *et al.*, 2002; Pellerito & Nagy, 2002). The present work is an extension of our interest in the structural aspects of organotin carboxylates (Parvez, Ali, Mazhar, Bhatti & Khokhar, 1999; Parvez, Ali, Bhatti *et al.*, 1999; Parvez, Ali, Mazhar, Bhatti & Choudhary, 1999; Parvez *et al.*, 2000, 2002). In this paper we report the structure of the title polymer, (I).



(I)

The structure of (I) is composed of two independent monomers (Figs. 1 and 2) in the asymmetric unit, forming polymeric chains (Fig. 3) lying parallel to the *b* axis that are separated by one-half unit-cell length along the *a* axis. In each polymeric chain, both O atoms of the acid moiety are bonded to Sn atoms with significantly different Sn—O distances, *viz.* Sn1—O1 2.129 (2) and Sn1—O2^{i} 2.578 (3) Å for one and

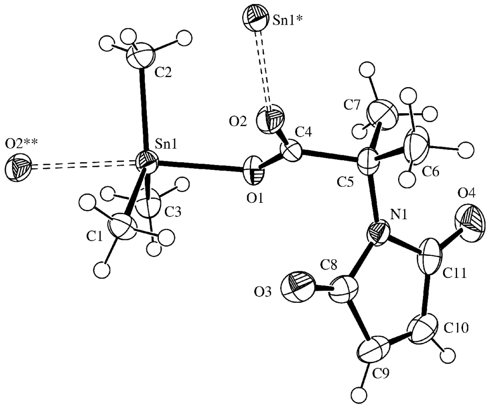


Figure 1
*ORTEP*II (Johnson, 1976) drawing of part of one polymeric chain of (I), with displacement ellipsoids drawn at the 50% probability level. Symmetry codes: (*) $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$; (**) $1 - x, y + \frac{1}{2}, \frac{1}{2} - z$.

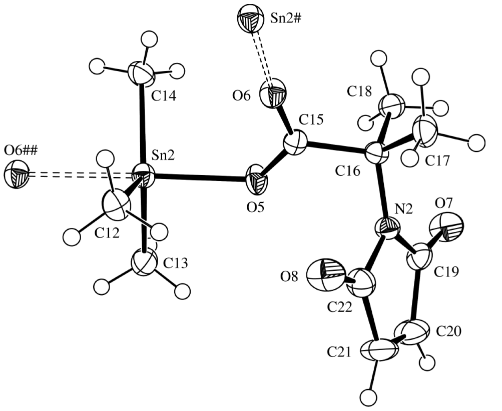


Figure 2
Drawing of part of the second polymeric chain of (I), with displacement ellipsoids drawn at the 50% probability level. Symmetry codes: (#) $-x, y + \frac{1}{2}, \frac{1}{2} - z$; (##) $-x, y - \frac{1}{2}, \frac{1}{2} - z$.

Sn2—O5 2.159 (2) and Sn2—O6^{ii} 2.508 (3) Å (symmetry codes as in Table 1) for the other polymer. The shorter Sn—O distances are indicative of covalent bonds and the longer distances clearly represent dative bonds. It is interesting to note that the shorter and longer Sn—O distances in the two polymers are significantly different from each other as well. However, these distances are in agreement with the corresponding values found for similar Sn complexes (Parvez, Ali, Mazhar, Bhatti & Khokhar, 1999; Parvez, Ali, Bhatti *et al.*, 1999; Parvez, Ali, Mazhar, Bhatti & Choudhary, 1999; Parvez *et al.*, 2000, 2002). The geometry around each Sn atom is distorted trigonal-bipyramidal, with three methyl groups occupying the equatorial positions. The Sn—C distances (Table 1) are identical within 3σ limits, with the exception of Sn2—C12, which lies within 4σ limits of the rest of the Sn—C distances [mean Sn—C = 2.117 (2) and 2.121 (7) Å for the two polymers]. Atoms Sn1 and Sn2 lie 0.202 (2) and 0.164 (2) Å, respectively, out of the equatorial planes formed by the three methyl C atoms, towards the more strongly bonded O atoms, a feature commonly observed in Sn–carboxylate complexes (Parvez, Ali, Mazhar, Bhatti & Khokhar, 1999; Parvez, Ali, Bhatti *et al.*, 1999; Parvez, Ali, Mazhar, Bhatti & Choudhary, 1999; Parvez *et al.*, 2000, 2002). In both polymer chains, the O—Sn—O angles are close to linear [170.46 (9) and 171.92 (9)°], the C—Sn—C angles lie between 111.22 (17) and 123.91 (16)°, deviating from the ideal value of 120°, and the O—Sn—C angles are in the range 81.03 (1)–98.56 (13)°. The molecular dimensions in the ligand are normal.

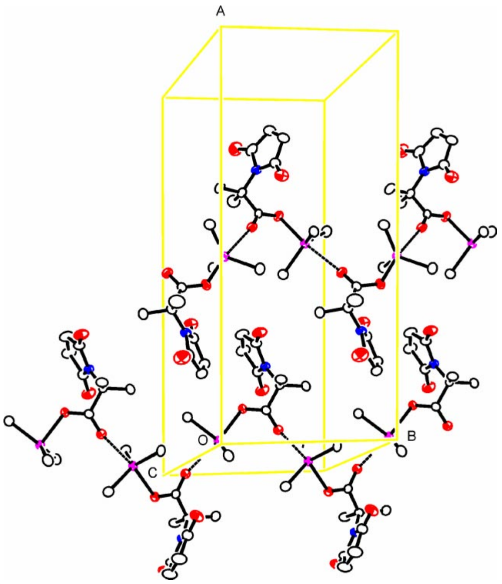


Figure 3
Two polymeric chains of (I). H-atoms have been omitted for clarity.

Experimental

The ligand was prepared by stirring equimolar quantities of maleic anhydride and α-aminoisobutyric acid in acetic acid for 12 h. following the procedure reported earlier (Rich *et al.*, 1975). The 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 continuously removed *via* a Dean–Stark apparatus. After cooling, toluene was removed from the orange layer, trimethyltin(IV) chloride (0.01 mol) was added in toluene, 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

$[Sn(C_8H_8NO_4)(CH_3)_3]$
$M_r = 345.95$
Monoclinic, $P2_1/c$
$a = 21.738 (5)$ Å
$b = 9.700 (2)$ Å
$c = 13.447 (3)$ Å
$\beta = 102.442 (13)°$
$V = 2768.8 (11)$ Å^3
$Z = 8$
$D_x = 1.660$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 11788 reflections
$\theta = 3.4$–$27.5°$
$\mu = 1.85$ mm^{-1}
$T = 173 (2)$ K
Block, colorless
0.18 × 0.15 × 0.15 mm

Data collection

Nonius KappaCCD diffractometer
ω and φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
$T_{min} = 0.725$, $T_{max} = 0.761$
11788 measured reflections
6332 independent reflections
4188 reflections with $I > 2\sigma(I)$
$R_{int} = 0.055$
$\theta_{max} = 27.5°$
$h = -28 \rightarrow 28$
$k = -12 \rightarrow 12$
$l = -17 \rightarrow 17$

Acta Cryst. (2004). E**60**, m554–m556 Masood Parvez *et al.* • $[Sn(C_8H_8NO_4)(CH_3)_3]$ **m555**

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.081$
$S = 0.96$
6332 reflections
307 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.026P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.85$ e Å^{-3}
$\Delta\rho_{\min} = -0.81$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—C2	2.114 (4)	Sn2—C14	2.115 (4)
Sn1—C3	2.119 (4)	Sn2—C13	2.118 (4)
Sn1—C1	2.119 (4)	Sn2—C12	2.131 (4)
Sn1—O1	2.129 (2)	Sn2—O5	2.159 (2)
Sn1—O2^{i}	2.578 (3)	Sn2—O6^{ii}	2.508 (3)
O1—C4	1.290 (4)	O5—C15	1.273 (4)
O2—C4	1.226 (4)	O6—C15	1.240 (4)
O3—C8	1.201 (4)	O7—C19	1.199 (5)
O4—C11	1.208 (5)	O8—C22	1.208 (5)
N1—C8	1.405 (5)	N2—C22	1.394 (5)
N1—C11	1.405 (5)	N2—C19	1.405 (5)
N1—C5	1.483 (5)	N2—C16	1.476 (4)
C2—Sn1—C3	113.90 (17)	C14—Sn2—C13	123.32 (16)
C2—Sn1—C1	123.91 (16)	C14—Sn2—C12	123.65 (15)
C3—Sn1—C1	119.48 (16)	C13—Sn2—C12	111.22 (17)
C2—Sn1—O1	97.74 (13)	C14—Sn2—O5	94.44 (13)
C3—Sn1—O1	89.59 (12)	C13—Sn2—O5	90.53 (13)
C1—Sn1—O1	98.56 (13)	C12—Sn2—O5	98.34 (12)
C2—Sn1—O2^{i}	89.08 (13)	C14—Sn2—O6^{ii}	83.62 (12)
C3—Sn1—O2^{i}	83.66 (12)	C13—Sn2—O6^{ii}	82.81 (12)
C1—Sn1—O2^{i}	81.03 (12)	C12—Sn2—O6^{ii}	90.47 (12)
O1—Sn1—O2^{i}	171.92 (9)	O5—Sn2—O6^{ii}	170.46 (9)
C4—O1—Sn1	121.0 (2)	C15—O5—Sn2	123.5 (2)
C8—N1—C11	108.5 (3)	C22—N2—C19	109.8 (3)
C8—N1—C5	123.2 (3)	C22—N2—C16	119.7 (3)
C11—N1—C5	127.1 (4)	C19—N2—C16	130.5 (3)

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

The H atoms were located in a difference Fourier synthesis and were included in the refinement at idealized positions with $U_{\rm iso}$ = 1.5 (methyl) and 1.2 (methylene) times $U_{\rm eq}$ of the atoms to which they are bonded, with C—H 0.98 and 0.95 Å for methyl and methylene types, respectively. The final difference map was free of any chemically significant features.

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.

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m556 Masood Parvez *et al.* • $[Sn(C_8H_8NO_4)(CH_3)_3]$ *Acta Cryst.* (2004). E**60**, m554–m556