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catena-Poly[[tri-*n*-butyltin(IV)]-μ-cyclopent-2-enylacetato-κ^2O:O']

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Key indicators

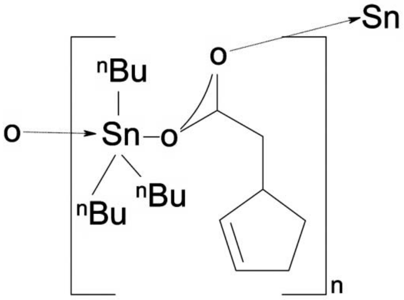
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.007 Å
R factor = 0.028
wR factor = 0.062
Data-to-parameter ratio = 23.6

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

The title compound, $[Sn(C_4H_9)_3(C_7H_9O_2)]_n$, forms a polymeric chain involving both O atoms of the carboxylate group. The Sn atom is five-coordinate and shows a distorted trigonal–bipyramidal geometry wherein the Cα atoms of the *n*-butyl chains occupy the equatorial positions. Two O atoms from symmetry-related ligands are asymmetrically bonded to the Sn atom and occupy axial positions with significantly different Sn—O distances of 2.209 (2) and 2.358 (2) Å.

Comment

The structural chemistry of organotin compounds in general and organotin carboxylates in particular has gained considerable interest over the past 30 years (Tiekink, 1991, 1994; Gielen, 1996; Mazhar *et al.*, 2001). The syntheses and biological activities of a wide variety of organotin complexes have been reported (Davies & Smith, 1982; Magos, 1986; Tagliavini, 1992; Ronconi *et al.*, 2002). Several organotin complexes have been actively investigated as potential antitumor agents (Crowe, 1987; Penninks, 1990). Continuing our interest in this important field (Parvez, Ali, Mazhar, Bhatti & Khokhar, 1999; Parvez, Ali, Bhatti *et al.*, 1999; Parvez, Ali, Mazhar, Bhatti & Choudhary, 1999; Parvez *et al.*, 2000, 2002; Sadiq-ur-Rehman, Shouldice *et al.*, 2004; Sadiq-ur-Rehman, Abdelrahman *et al.*, 2004), we report the structure of the title compound, (I).



(I)

The Sn atom in the structure of (I) (Fig. 1) is bonded to three *n*-butyl groups in equatorial positions with almost identical Sn—C distances [mean distance 2.141 (7) Å]. The axial positions are occupied by O atoms derived from two carboxylate ligands with a nearly linear O—Sn—O angle (Table 1). The O atoms from symmetry-related ligands are asymmetrically bonded to the Sn atom with Sn1—O1 shorter than Sn1—O2^i (Table 1). The geometry around the Sn atom is trigonal bipyramidal, with the Sn atom 0.122 (2) Å out of the plane defined by the Cα atoms towards the more strongly bound O atom. It is interesting to note that the C—O distances

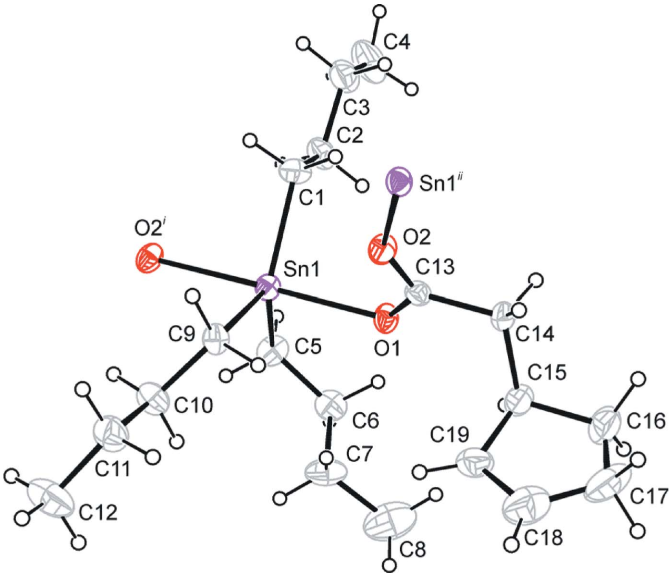


Figure 1
Part of the polymeric structure in (I), showing displacement ellipsoids plotted at the 30% probability level. Symmetry codes are as in Table 1.

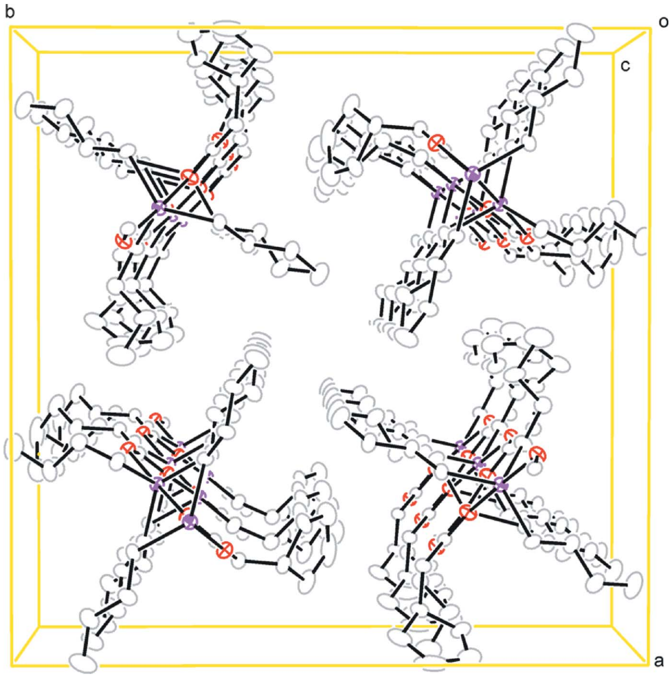


Figure 2
The packing for (I), viewed down the *c* axis.

within a given ligand, *i.e.* O1—C13 and O2—C13 (Table 1), are intermediate between a single and a double bond, and, therefore, represent a delocalized system. The structure is composed of infinite chains running parallel to the *c* axis, as indicated in Fig. 2.

Experimental

Bis(tributyltin) oxide (3.0 g, 5.03 mmol) and 2-(cyclopentenyl)acetic acid (1.27 g, 10.07 mmol) were refluxed for 8 h in dry toluene (100 ml) in a single-necked round-bottomed flask (250 ml). The water formed during the condensation reaction was removed *via* a Dean and Stark apparatus. After cooling the reaction mixture to room temperature, the solvent was removed under vacuum. The product thus obtained was recrystallized from a mixture of chloroform:*n*-hexane (9:1), which on slow evaporation at room temperature gave crystals suitable for the X-ray crystallographic study.

Crystal data

$[Sn(C_4H_9)_3(C_7H_9O_2)]$
$M_r = 415.17$
Tetragonal, $I\bar{4}$
$a = 20.559$ (6) Å
$c = 10.053$ (7) Å
$V = 4249$ (3) Å^3
$Z = 8$

$D_x = 1.298$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 1.21$ mm^{-1}
$T = 173$ (2) K
Block, colorless
$0.20 \times 0.10 \times 0.09$ mm

Data collection

Nonius KappaCCD diffractometer
ω and φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
$T_{min} = 0.794$, $T_{max} = 0.899$

8643 measured reflections
4776 independent reflections
4277 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.062$
$S = 1.04$
4776 reflections
202 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.024P)^2 + 2.15P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.42$ e Å^{-3}
Absolute structure: Flack (1983), 2198 Friedel pairs
Flack parameter: −0.02 (2)

Table 1
Selected geometric parameters (Å, °).

Sn1—C1	2.131 (4)	Sn1—O2^i	2.358 (2)
Sn1—C5	2.146 (3)	O1—C13	1.278 (4)
Sn1—C9	2.147 (3)	O2—C13	1.249 (4)
Sn1—O1	2.209 (2)		
C1—Sn1—C5	116.17 (16)	C5—Sn1—O2^i	81.83 (12)
C1—Sn1—C9	124.25 (14)	C9—Sn1—O1	95.20 (11)
C1—Sn1—O1	92.47 (13)	C9—Sn1—O2^i	90.45 (11)
C5—Sn1—O1	92.02 (12)	O1—Sn1—O2^i	173.13 (8)
C1—Sn1—O2^i	87.54 (13)	C13—O1—Sn1	119.1 (2)
C5—Sn1—C9	118.61 (15)	C13—O2—Sn1^{ii}	141.31 (19)

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

H atoms were included in the refinement at geometrically idealized positions, C—H = 0.95–1.00 Å and $U_{iso}(H) = 1.2U_{eq}$(parent atom).

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SAPI91* (Fan, 1991); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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