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catena-Poly[tributyltin(IV)-μ-acetato]

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.016 Å
Disorder in main residue
R factor = 0.053
wR factor = 0.060
Data-to-parameter ratio = 19.0

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

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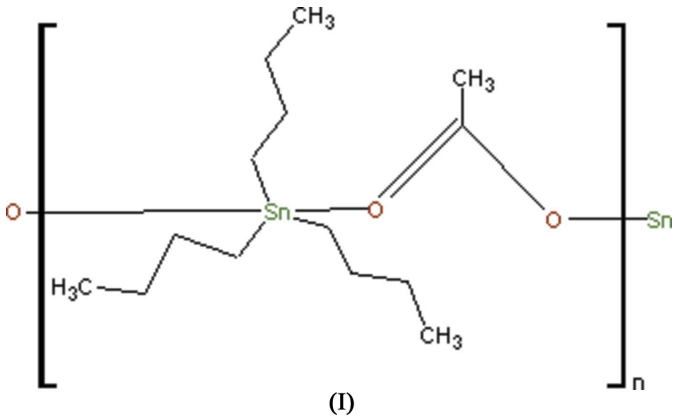
The title compound, $[Sn(C_4H_9)_3(C_2H_3O_2)]_n$, forms characteristic polymeric chains, in which Sn atoms are bridged by acetate groups, bonding through both O atoms. The coordination geometry of the Sn atom is slightly distorted trigonal bipyramidal. The butyl groups occupy the equatorial positions, with Sn—C bond lengths in the range 2.115 (10)–2.129 (9) Å, while the axial Sn—O bond lengths are 2.178 (5) and 2.422 (5) Å.

Comment

The structural chemistry of organotin compounds that have a coordination number greater than four has been studied essentially because of the biological activity, enhanced reactivity and stereochemical non-rigidity of these compounds (Mehring *et al.*, 1998). Triorganostannyl esters generally have low solubility in organic solvents because of their polymeric structures. Dilution of triorganotin carboxylates in organic solvents usually produces oligomeric and monomeric species containing tetrahedral tin atoms and free ester carbonyl groups (McFarlane & Wood, 1972; Simons & Graham, 1967). Although tributyltin compounds are only active against Gram-positive bacteria (Schumann & Roth, 1968; Van Der Kerk & Luijten, 1954), their combination with a second chemical which combats Gram-negative species produces highly effective disinfectants which may be used on open areas posing a risk of infection, such as hospital floors and in sports pavilions. One such formulation contains a mixture of tributyltin benzoate and formaldehyde (Killian & Wrackmeyer, 1977). Organotin(IV) complexes with ligands having fluorine, sulfur, nitrogen and oxygen as substituents have been widely tested for their possible use in cancer chemotherapy and a few have proved to be active against tumours (Gielen *et al.*, 1995). Furthermore, it has been shown that trialkyltin carboxylates exhibit greater antitumour activity, lower mammalian toxicity and less nephrotoxicity than *cis*-platin (Sandhu *et al.*, 1992; Danish *et al.*, 1996; Gielen *et al.*, 1992). Continuing our interest in the structural aspects of organotin carboxylates (Saeed *et al.*, 2005), we now report the synthesis and crystal structure of the title compound, (I).

A view of (I) is shown in Fig. 1. The structure consists of Sn atoms bridged by acetate groups, through both O atoms, to form polymeric chains, giving significantly different Sn—O distances [Sn1—O1Ac1 = 2.422 (5) Å and Sn1—O1Ac2 = 2.178 (5) Å]. The coordination geometry of the Sn atom is distorted trigonal-bipyramidal with the three butyl groups occupying the equatorial positions. The Sn—C distances are the same within experimental error [Sn1—C1 = 2.129 (9) Å, Sn1—C5 = 2.128 (8) Å and Sn1—C9 = 2.115 (10) Å]. The O1Ac1—Sn1—O1Ac2 angle is 172.0 (2)° and the C—Sn—C

angles range from 116.5 (4) to 123.2 (4)°, deviating little from the ideal value of 120°. The O—Sn—C angles are in the range 88.7 (3)–93.6 (3) °.



(I)

Experimental

Tributyltin(IV) chloride (3.97 g, 3.07 mmol, from Aldrich) and sodium acetate (1 g, 1.22 mmol, from Aldrich) were dissolved in dry toluene (100 ml) in a two-necked round-bottomed flask. The reaction mixture was refluxed for 24 h with continuous stirring. The resulting sodium salt was removed by filtration and the solvent was evaporated under reduced pressure. The resulting solid material was recrystallized from a mixture of toluene and *n*-hexane (95:5) giving fine colourless crystals (yield 82%, m.p. 354 K).

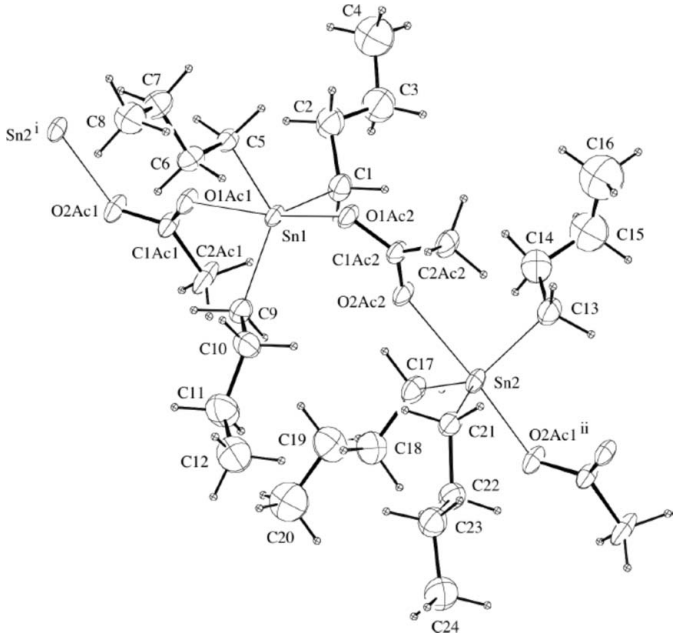


Figure 1
ORTEPII (Johnson, 1976) plot of the title compound, with displacement ellipsoids drawn at the 10% probability level. H atoms are drawn as spheres of arbitrary radius. Only one disorder component is shown. [Symmetry codes: (i) $1 + x, y, z$; (ii) $x - 1, y, z$.]

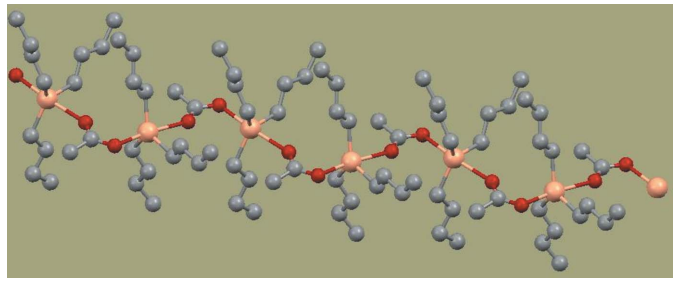


Figure 2
View of part of the polymeric structure of (I). Only one disorder component is shown and H atoms have been omitted.

Crystal data

$[Sn(C_4H_9)_3(C_2H_3O_2)]$	$D_x = 1.29$ Mg m^{-3}
$M_r = 349.1$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 10 reflections
$a = 10.386$ (4) Å	$\theta = 11$–$12°$
$b = 20.924$ (3) Å	$\mu = 1.42$ mm^{-1}
$c = 16.584$ (6) Å	$T = 294$ K
$\beta = 92.87$ (2)°	Prism, colourless
$V = 3599$ (2) Å^3	0.30 × 0.10 × 0.06 mm
$Z = 8$	

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.015$
ω–2θ scans	$\theta_{max} = 25°$
Absorption correction: analytical (de Meulenaer & Tompa, 1965)	$h = 0 \rightarrow 12$
$T_{min} = 0.84$, $T_{max} = 0.93$	$k = 0 \rightarrow 24$
6704 measured reflections	$l = -19 \rightarrow 19$
6332 independent reflections	1 standard reflections
3844 reflections with $I > 2\sigma(I)$	frequency: 30 min
	intensity decay: 28%

Refinement

Refinement on F	H-atom parameters not refined
$R[F^2 > 2\sigma(F^2)] = 0.053$	$w = 1/[\sigma^2(F) + 0.0004F^2]$
$wR(F^2) = 0.060$	$(\Delta/\sigma)_{max} = 0.009$
$S = 1.87$	$\Delta\rho_{max} = 0.91$ e Å^{-3}
3844 reflections	$\Delta\rho_{min} = -0.90$ e Å^{-3}
202 parameters	

All C—H distances were fixed at 1.0 Å with $U_{iso}(H) = U_{eq}(C)$. A number of the *n*-butyl C atoms, especially the terminal C atoms, were difficult to locate and refine, and clearly were affected by high thermal motion and disorder. Each of these (C4/C4′, C8/C8′,C15/C15′, C16/C16′ and C20/C20′) was refined as disordered over two sites with equal occupancy using isotropic displacement parameters. A linear correction for decomposition was applied, which accounts for the large intensity decay.

Data collection: *CAD-4 Software* (Schagen *et al.*, 1989); cell refinement: *CAD-4 Software*; data reduction: local program; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *RAELS* (Rae, 1996); molecular graphics: *ORTEPII*, (Johnson, 1976); software used to prepare material for publication: local programs.

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