Tetrakis[μ-3-(4-methylbenzoyl)propionato]-κ⁴O:O′;κ⁴O:O-di-μ₃-oxo-tetrakis[dibutyltin(IV)]

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The centrosymmetric title compound, $[Sn_4(C_4H_9)_8\text{-}(C_{11}H_{11}O_3)_4O_2]$, contains a planar Sn_2O_2 core with the O atoms bonded to two dibutylbis[3-(4-methylbenzoyl)-propionato]tin units. The exocyclic Sn atom is five-coordinate with a distorted C_2SnO_3 trigonal–bipyramidal geometry and the endocyclic Sn atom is six-coordinate with a C_2SnO_4 skew-trapezoidal bipyramidal geometry.

Comment

1,1,3,3-Tetraorgano-1,3-dicarboxylatodistannoxanes, $R_2(R'\text{-}CO_2)SnOSn(O_2CR')R_2$, obtained from the 1:1 reaction of R_2SnO and $R'CO_2H$, are usually dimeric (Chandrasekhar et al., 2002). Tiekink (1991, 1994) has classified these compounds into four major types, the most common of which contains two bridging and two monodentate carboxylate groups, with one five-coordinate and one six-coordinate Sn atom. The title compound, (I), belongs to this structural type.

(I)
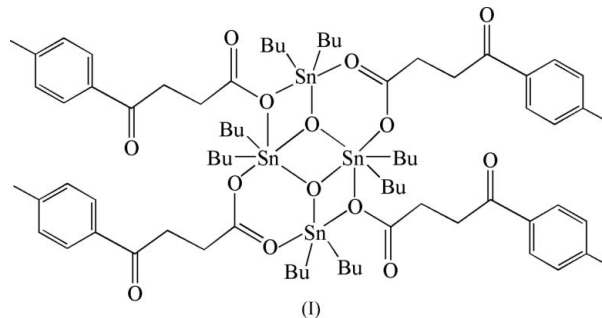
In the crystal structure, the molecule of (I) lies on a centre of inversion (Fig. 1). The endocyclic Sn atoms of the planar Sn_2O_2 core are six-coordinate with a C_2SnO_4 skew-trapezoidal bipyramidal geometry, in which Sn1 is coordinated by two tridentate bridging O atoms, two 3-(4-methylbenzoyl)-propionate anions and two C atoms of the n-butyl groups. The endocyclic Sn—O distances [Sn1—O1 and Sn1—O1ⁱ; symmetry code: (i) $1 - x$, $1 - y$, $-z$] in the Sn_2O_2 core are 2.166 (2) and 2.042 (2) Å, respectively, and the exocyclic Sn1—O5 and Sn1—O2 distances are respectively relatively short [2.295 (3) Å] and very long [2.713 (3) Å]. Similar arrangements have been reported for the tetrabutylbis[(N,N-diethylthiocarbamoylthio)acetato]distannoxane dimer (Ng & Kumar Das, 1995), the tetrabutylbis(N-phthaloylglycinato)-distannoxane dimer (Parvez et al., 2000), the tetrabutylbis(N-phthaloylphenylalaninato)distannoxane dimer (Hans et al., 2002) and bis[tetrabutylbis(4-(4-biphenyl)-4-oxobutanoato)-distannoxane] (Tian et al., 2005).

The exocyclic Sn2 atom is five-coordinate with a distorted trigonal–bipyramidal geometry, in which the axial positions are occupied by O atoms (O1 and O6^i) of two carboxylates [O1—Sn2—O6^i = 167.93 (12)°]. Atom Sn2 lies 0.0918 (3) Å out of the trigonal plane defined by O1, C9 and C13, in the direction of atom O2. The three Sn—O distances around Sn2 are in the range 2.020 (2)–2.268 (3) Å, while the Sn2⋯O3 distance of 3.023 (3) Å indicates only a weak interaction between these two atoms. The Sn—C distances lie within the narrow range 2.095 (5)–2.137 (6) Å, in agreement with the values observed in the comparable complexes mentioned above.

Experimental

Di-*n*-butyl oxide (0.373 g, 1.5 mmol) and 4-(4-methylphenyl)-4-oxobutanoic acid (0.288 g, 1.5 mmol) in 60 ml of benzene were refluxed for 4 h with azeotropic removal of water *via* a Dean–Stark trap. The resulting clear solution was evaporated under vacuum and the resulting white crystalline material was recrystallized from ethanol. The product (yield 81%, m.p. 382–383 K) was then dissolved in methanol, and colourless crystals were grown by slow evaporation. Elemental analysis found: C 52.56, H 6.47%; calculated: C 52.81, H 6.76%.

Crystal data

[Sn$_4$(C$_4$H$_9$)$_8$(C$_{11}$H$_{11}$O$_3$)$_4$O$_2$]	V = 2034.1 (4) Å^3
M_r = 1728.45	Z = 1
Triclinic, $P\bar{1}$	D_x = 1.411 Mg m^{-3}
a = 12.0914 (15) Å	Mo $K\alpha$ radiation
b = 12.2993 (16) Å	μ = 1.27 mm^{-1}
c = 15.0996 (19) Å	T = 295 (2) K
α = 70.392 (2)°	Block, colourless
β = 87.612 (2)°	0.33 × 0.18 × 0.14 mm
γ = 74.361 (2)°	

Data collection

Bruker SMART APEX CCD diffractometer	11699 measured reflections
φ and ω scans	7849 independent reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)	6090 reflections with $I > 2\sigma(I)$
T_{min} = 0.679, T_{max} = 0.842	R_{int} = 0.013
	θ_{max} = 26.0°

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0605P)^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.037	$+ 0.6267P]$
$wR(F^2)$ = 0.110	where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max}$ = 0.001
7849 reflections	$\Delta\rho_{max}$ = 0.96 e Å^{-3}
440 parameters	$\Delta\rho_{min}$ = −0.49 e Å^{-3}
H-atom parameters constrained	

The C—C bonds and 1,3-distances of the *n*-butyl groups were restrained to 1.52 (1) and 2.50 (2) Å, respectively. The four C atoms of two *n*-butyl groups are disordered over two positions; their site
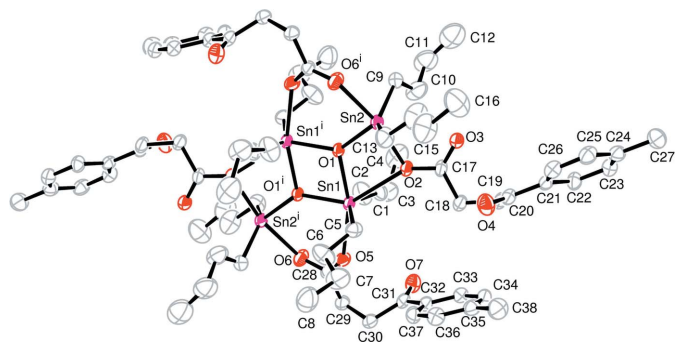
Figure 1
The molecular structure of (I), showing displacement ellipsoids at the 20% probability level. H atoms and the minor components, C3′, C4′, C7′ and C8′, of the disordered atoms in the *n*-butyl groups have been omitted [symmetry code: (i) $1 - x, 1 - y, -z$].

occupancies were refined to 0.741 (14):0.259 (14) for C3/C3′ and C4/C4′, and 0.574 (9):0.426 (9) for C7/C7′ and C8/C8′. H atoms were placed at calculated positions and were included in the refinement in the riding-model approximation, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C) for aromatic H atoms, C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms, and C—H = 0.97 Å and U_{iso}(H) = 1.2U_{eq}(C) for methylene H atoms.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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