metal-organic papers

Octabutyl-1κ^2C,2κ^2C,3κ^2C,4κ^2C-di-μ_2-3,5-dinitrobenzoato-1:2κ^2O:O';3:4κ^2O:O'-bis-(3,5-dinitrobenzoato)-1κO,4κO-di-μ_3-oxo-1:2:3κ^3O:O:O;2:3:4κ^3O:O:O-tetratin(IV)

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.014 Å
R factor = 0.048
wR factor = 0.149
Data-to-parameter ratio = 14.6

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

The title compound, [Sn$_4$(C$_4$H$_9$)$_8$(C$_7$H$_3$N$_2$O$_6$)$_4$O$_2$], is a cluster built up by inversion symmetry around the central Sn$_2$O$_2$ ring. Both unique SnO$_3$C$_2$ centres have distorted trigonal–bipyramidal geometry with O atoms in the axial positions.

Comment

The title compound, (I) (Fig. 1), is a cluster containing four Sn atoms and a total of 98 non-H atoms. The whole molecule is centrosymmetric with a central Sn$_2$O$_2$ core; the structure is similar to those of related compounds (Yin *et al.*, 2003). The μ_3-bridging O13 atom in the Sn$_2$O$_2$ ring is also attached to a Bu$_2$Sn unit. In addition, the C1-carboxylate group coordinates to two Sn atoms in a bridging mode. The C1—O1 and C2—O2 carboxylate bond lengths are very different (Table 1).

(I)
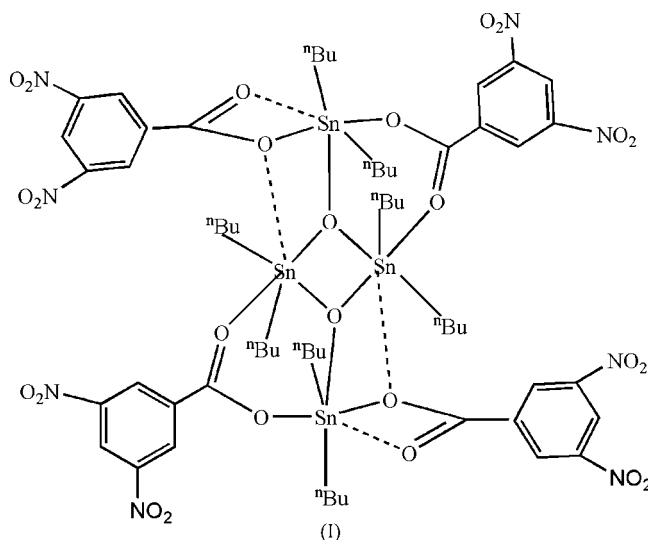
The geometries of both the Sn atoms are distorted trigonal–bipyramidal. For the exocyclic Sn1 species, atoms O1 and O7 are in axial positions [O1—Sn1—O7 = 169.4 (2)°] and the C atoms of the two butyl groups and O13 are in equatorial positions. The sum of the equatorial C—Sn—C and O—Sn—C angles is 359.8°, indicating approximate coplanarity for these atoms.

The geometry around the endocyclic atom Sn2 is slightly different from that of Sn1. Here, O2 and O13^i [symmetry code: (i) −x + 2, −y + 1, −z] are in axial positions [O13—Sn2—O2 = 162.4 (2)°] and the C atoms of the two butyl groups and O13 are in equatorial positions. The sum of the equatorial C—Sn—C and O—Sn—C angles is 343.4°, indicating a significant distortion from coplanarity for these atoms. This distortion may arise because of a short Sn2···O7^i contact of 2.815 (6) Å

(sum of the van der Waals radii = 4.0 Å; Vollano et al., 1984). A short Sn1···O8 contact of 2.949 (6) Å is also present (Fig. 2).

Experimental

A mixture of dibutyltin oxide (0.4978 g, 2.0 mmol) and 3,5-dinitro-benzoic acid (0.4242 g, 2.0 mmol) in methanol (80 ml) was heated under reflux for 8 h. The resulting clear solution was evaporated under vacuum. The product was crystallized from a mixture of dichloromethane/ethanol (1:1) giving blocks of (I) (yield 0.6419 g, 71%; m.p. 426 K). Analysis calculated for $C_{60}H_{84}N_8O_{26}Sn_4$: C 39.85, H, 4.68; N 6.20%; found: C 39.87, H 4.71, N, 6.23%.

Crystal data

$[Sn_4(C_4H_9)_8(C_7H_3N_2O_6)_4O_2]$
$M_r = 1808.11$
Triclinic, $P\bar{1}$
$a = 10.705$ (2) Å
$b = 13.333$ (3) Å
$c = 14.360$ (3) Å
$\alpha = 68.892$ (3)°
$\beta = 78.297$ (3)°
$\gamma = 80.285$ (3)°
$V = 1862.0$ (7) Å^3

$Z = 1$
$D_x = 1.613$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3347 reflections
$\theta = 2.4–25.0°$
$\mu = 1.41$ mm^{-1}
$T = 298$ (2) K
Block, colourless
$0.46 \times 0.40 \times 0.37$ mm

Data collection

Bruker SMART CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 1998)
$T_{min} = 0.564$, $T_{max} = 0.624$
9788 measured reflections

6473 independent reflections
4391 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 25.0°$
$h = -11 \rightarrow 12$
$k = -15 \rightarrow 15$
$l = -17 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.149$
$S = 1.00$
6473 reflections
442 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0672P)^2 + 5.5003P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.03$ e Å^{-3}
$\Delta\rho_{min} = -0.93$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—O13	2.021 (5)	Sn2—C27	2.127 (8)
Sn1—C19	2.116 (8)	Sn2—O13^i	2.151 (5)
Sn1—C15	2.118 (9)	Sn2—O2	2.277 (5)
Sn1—O7	2.202 (5)	Sn2—O7^i	2.815 (6)
Sn1—O1	2.281 (5)	C1—O1	1.238 (9)
Sn1—O8	2.949 (6)	C1—O2	1.288 (9)
Sn2—O13	2.055 (5)	C8—O7	1.290 (10)
Sn2—C23	2.123 (8)	C8—O8	1.224 (10)
Sn1—O13—Sn2	134.2 (3)	Sn2—O13—Sn2^i	103.7 (2)
Sn1—O13—Sn2^i	121.8 (2)		

Symmetry code: (i) $-x+2, -y+1, -z$.

H atoms were positioned geometrically [C—H = 0.93 (CH), 0.97 (CH$_2$) and 0.96 Å (CH$_3$)] and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(methyl C). The highest peak is located 0.92 Å from atom Sn1.
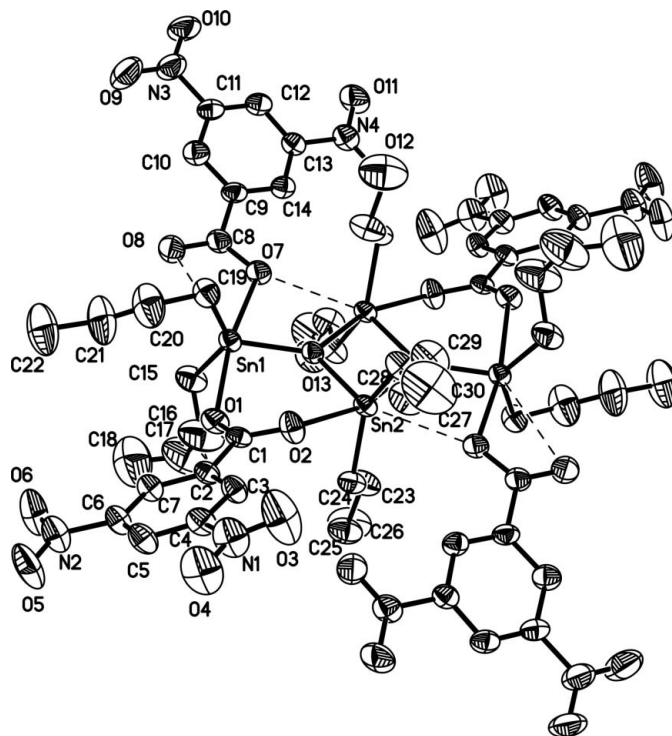

Figure 1
The molecular structure of (I), with 30% displacement ellipsoids (H atoms have been omitted for clarity). The unlabelled atoms are generated by the symmetry code $(2 - x, 1 - y, -z)$. Dashed lines indicate short Sn···O contacts.
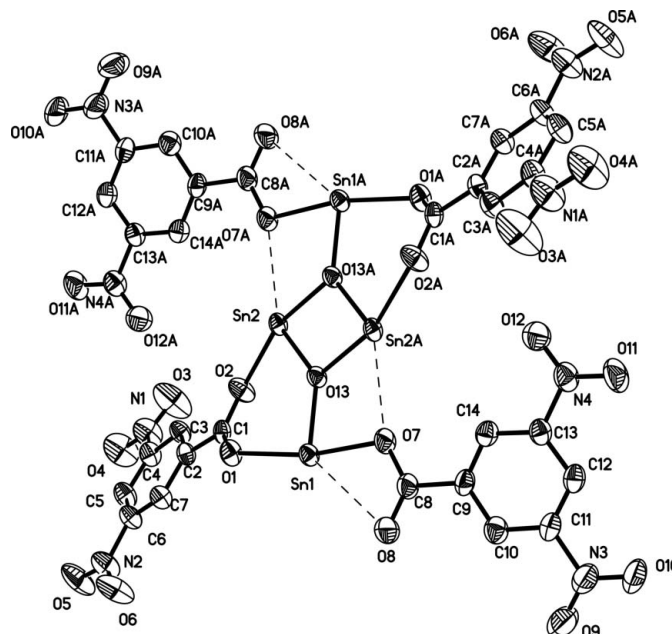

Figure 2
The molecular structure with the n-butyl groups omitted for clarity. Atoms with the suffix a are generated by the symmetry code $(2 - x, 1 - y, -z)$. Dashed lines indicate short Sn···O contacts

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1998); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine

structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXL97*.

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