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Octa-*n*-butylbis(μ_2-4-chloro-3,5-dinitrobenzoato-κ^2O:O')bis(4-chloro-3,5-dinitrobenzoato-κO)di-μ_3-oxo-tetratin(IV)

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Key indicators

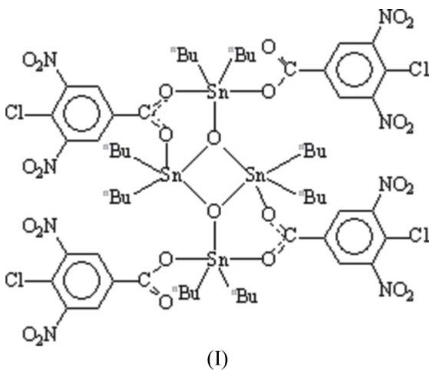
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.032
wR factor = 0.086
Data-to-parameter ratio = 15.3

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

The title compound, $[Sn_4O_2(C_7H_2ClN_2O_6)_4(C_4H_9)_8]$, is a centrosymmetric dimer of an oxoditin(IV) complex. Two dibutylbis[4-chloro-3,5-dinitrobenzoate]tin(IV) units containing monodentate and bridging bidentate carboxylate ligands are connected to the central Sn_2O_2 core. Each Sn atom adopts a distorted trigonal–bipyramidal geometry, with Sn—C distances lying in the narrow range 2.119 (3)–2.135 (3) Å, while Sn—O distances range between 2.037 (2) and 2.286 (2) Å.

Comment

There have been several reports dealing with the impact of organotin chemistry in the biosphere (Gielen, 1994; Ng *et al.*, 1991). Exploration of the structure–activity relationships of such systems has led to numerous reports in recent years (Gielen, 1994; Selvaratnam *et al.*, 1994; McManus *et al.*, 1994). The structural chemistry of organotin carboxylic acid esters has been extensively explored because of the rich diversity of structural motifs (Tiekink, 1994). Among organotin carboxylates, dimeric distannoxanes comprise the most interesting class with respect to their structural chemistry. Reports on crystallographic studies show that these compounds may adopt a variety of structural modes depending on the nature of organic substituents at the Sn atom or carboxylate ligand (Danish *et al.*, 1996). There have been numerous crystallographic reports on these compounds describing their dimeric nature and there are at least five distinct structural types known for them (Tiekink, 1991). In continuation of our studies of organotin(IV) carboxylates (Sadiq-ur-Rehman *et al.*, 2006), we have synthesized the title compound (I), the crystal structure of which is reported here.



(I)

The structure of (I) is composed of a centrosymmetric dimer of oxoditin units (Fig. 1). The endocyclic Sn—O distance in the central core [Sn1—O7 of 2.286 (2) Å] and the endocyclic distance [Sn1—O1 = 2.188 (2) Å] are quite similar

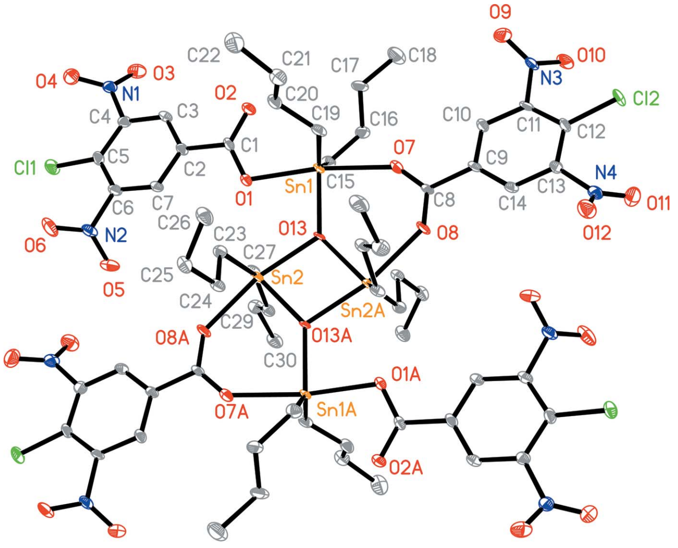


Figure 1
Structure of (I). Displacement ellipsoids are drawn at the 50% probability level. The atoms labelled with A are at the symmetry position (−*x*, −*y*, −*z*). H atoms have been omitted. For clarity, only one component of the disordered butyl group (C15–C18) is shown.

to those observed in the tetrabutylbis(*N*-phthaloylglycinato)distannoxane dimer (Parvez *et al.*, 2000) and the tetrabutylbis(*N*-phthaloylphenylalaninato)distannoxane dimer (Hans *et al.*, 2002). Both independent Sn atoms in (I) are in a five-coordinate O_3C_2Sn distorted trigonal–bipyramidal geometry. The carboxylate ligand shows different modes of coordination with Sn. Firstly, it acts as monodentate, coordinated to Sn1 *via* O1; the Sn1···O2 distance is 2.883 (2) Å, *i.e.* too long to be considered bonding [likewise, the Sn2···O1 distance of 2.913 (2) Å]. In the other coordination mode, the ligand bridges two Sn atoms in a bidentate fashion, thus resulting in a six-membered Sn1—O7—C8—O8—Sn2^{i}—O13 ring [symmetry code: (i) −*x*, −*y*, −*z*]. The Sn—C distances lie in the very narrow range 2.119 (3)–2.135 (3) Å, while the Sn—O distances range between 2.037 (2) and 2.286 (2) Å (Table 1).

Experimental

A mixture of 3,5-dinitro-4-chlorobenzoic acid (2 g, 1 mmol) and di-*n*-butyltin oxide (2.02 g, 1 mmol) was refluxed in dry toluene (150 ml) for 5–6 h using a Dean and Stark trap. The reaction mixture was cooled to room temperature and solvent was evaporated under reduced pressure. The solid product was recrystallized from chloroform, resulting in rod-shaped crystals of (I) (yield 85%, m.p. 513–514 K).

Crystal data

$[Sn_4O_2(C_7H_2ClN_2O_6)_4(C_4H_9)_8]$
M_r = 1945.88
Triclinic, $P\bar{1}$
a = 12.1784 (11) Å
b = 12.5143 (12) Å
c = 13.5682 (12) Å
α = 104.597 (1)°
β = 110.770 (1)°
γ = 95.960 (1)°

V = 1827.9 (3) Å^3
Z = 1
D_x = 1.768 Mg m^{-3}
Mo *K*α radiation
μ = 1.58 mm^{-1}
T = 100 (2) K
Rod, colourless
0.40 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.571, T_{max} = 0.743

14580 measured reflections
7352 independent reflections
6497 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 26.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.086
S = 1.02
7352 reflections
482 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0514P)^2 + 0.862P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.61 e Å^{-3}
$\Delta\rho_{min}$ = −1.47 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—O13	2.0391 (19)	Sn2—C23	2.119 (3)
Sn1—C19	2.124 (3)	Sn2—C27	2.125 (3)
Sn1—C15	2.135 (3)	Sn2—O13	2.1708 (19)
Sn1—O1	2.188 (2)	Sn2—O8^{i}	2.276 (2)
Sn1—O7	2.286 (2)	Sn2···Sn2^{i}	3.2908 (5)
Sn2—O13^{i}	2.0372 (19)		
O13—Sn1—C19	106.03 (10)	O13^{i}—Sn2—C23	108.48 (10)
O13—Sn1—C15	108.78 (10)	O13^{i}—Sn2—C27	114.33 (10)
C19—Sn1—C15	144.19 (12)	C23—Sn2—C27	136.69 (12)
O13—Sn1—O1	83.08 (8)	O13^{i}—Sn2—O13	77.16 (8)
C19—Sn1—O1	99.15 (10)	C23—Sn2—O13	97.19 (10)
C15—Sn1—O1	93.09 (10)	C27—Sn2—O13	98.24 (10)
O13—Sn1—O7	89.89 (8)	O13^{i}—Sn2—O8^{i}	91.86 (8)
C19—Sn1—O7	86.80 (10)	C23—Sn2—O8^{i}	89.20 (10)
C15—Sn1—O7	85.19 (11)	C27—Sn2—O8^{i}	83.31 (10)
O1—Sn1—O7	171.81 (8)	O13—Sn2—O8^{i}	168.58 (8)

Symmetry code: (i) −*x*, −*y*, −*z*.

H atoms were included in calculated positions using the riding model, with C—H = 0.95–0.99 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$, or $1.5U_{eq}(C)$ for methyl groups. In one of the butyl groups (C15–C18), atoms C16 and C17 are disordered over two sites (C16/C17 and C16*A*/C17*A*); the occupancies refined to 0.496 (15) and 0.504 (15), respectively, and were fixed at 0.5 for the final cycles of refinement. The highest residual density peak is located 0.95 Å from atom Sn2 and the deepest hole is located 0.98 Å from atom Sn1.

Data collection: *SMART* (Bruker, 2001); 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: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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