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μ-3,4,5,6-Tetrafluorophthalato-bis[tris(2-methyl-2-phenylpropyl)tin(IV)]

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.006 Å
R factor = 0.028
wR factor = 0.068
Data-to-parameter ratio = 19.9

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

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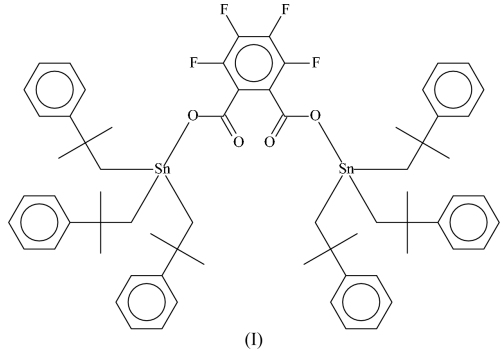
The molecule of the title compound, $[Sn_2(C_{10}H_{13})_6(C_8F_4O_4)]$, has crystallographic twofold symmetry. The Sn atoms are four-coordinate in a tetrahedral geometry.

Comment

The older structural literature on organotin(IV) carboxylates (Tiekink, 1991; 1994) lists no example of a carboxylate derived by condensing fenbutatin oxide, $\{C_6H_5C(CH_3)_2CH_2]_3Sn\}_2O$, an industrial miticide, with carboxylic acids. The first authenticated example of a tris(2-methyl-2-phenylpropyl)tin carboxylate is a four-coordinate phenoxyacetate (Bao *et al.*, 1998). The acetate is assigned a five-coordinate geometry arising from chelation by the acetate group (Bomfim *et al.*, 2002), but a comparison with the phenoxyacetate suggests that the dative linkage is probably inconsequential, so that the geometry is better interpreted as tetrahedral. An arylcarboxylate having an organogermyl substituent also shows unambiguous tetrahedral coordination (Fang *et al.*, 2001). Other carboxylates that were synthesized for an acaricidal screening trial are most likely monomeric tetrahedral compounds (Zhang *et al.*, 1999).



(I)

Tetrafluorophthalic acid, whose crystal structure has been reported (Gowda & Rudman, 1983), has not been condensed with organotin oxides/hydroxides, probably because of the difficulty of obtaining the reagent (Wen *et al.*, 1999). The acid is an important pharmaceutical intermediate used in the synthesis of antimicrobial drugs (Zhou & Zhang, 2000). A cursory search through the chemical literature revealed only a few examples of organometallic tetrafluorophthalates, for example, bis(tetraphenylantimony) tetrafluorophthalate (Sharutin *et al.*, 2002).

The tetrahedral nature of tris(2-methyl-2-phenylpropyl)tin carboxylates arises from crowding of the three organic groups covalently bonded to tin, and such compounds are an exception to the observation (Ng *et al.*, 1988) that trialkyltin carboxylates auto-associate into polymers through carboxyl-

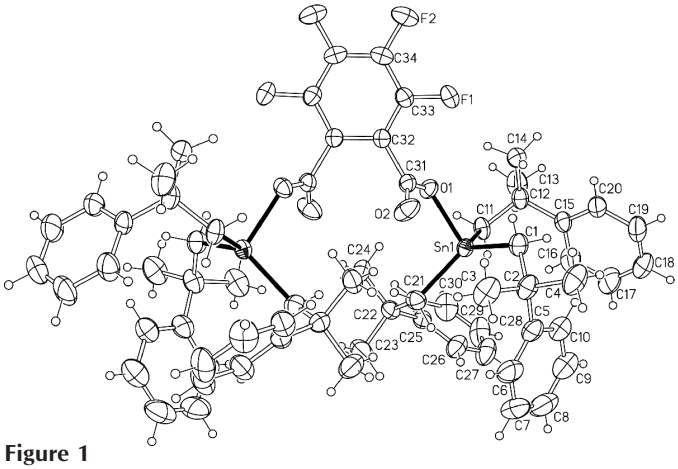


Figure 1
*ORTEP*II (Johnson, 1976) plot of (I), showing ellipsoids at the 30% probability level. H atoms are drawn as spheres of arbitrary radii. Unlabeled atoms are related to labeled atoms by $1 - x, y, -z$

ate bridging. Tetrahedral coordination is also observed in the present fluorine-substituted phthalate, whose carboxyl units bind to two triorganotin cations to furnish a dinuclear entity, (I) (Fig. 1). The molecule has crystallographic twofold symmetry. Bond dimensions, particularly the covalent Sn—O distance, are similar to those found in the carboxylate structures mentioned above (Table 1).

Experimental

The title compound was synthesized by condensing bis[tris(2-phenyl-2-methylpropyl)tin] oxide (2.64 g, 2.5 mmol) with 3,4,5,6-tetrafluorophthalic acid (0.60 g, 2.5 mmol) in benzene (60 ml). The water was removed using a Dean–Stark water separator and the condensation was complete in about 8 h. The compound was purified by recrystallization from ethanol, and crystals were obtained from a chloroform–cyclohexane (1:1 *v*/*v*) solution of the compound in 80% yield. Analysis found: C 63.86, H 6.07%; calculated for $C_{68}H_{78}F_4O_4Sn_2$: C 64.17, H 6.18%. IR (KBr disc): ν_{as}(COO) 1656, ν_s(COO) 1346 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.28–7.11 (30H, *m*, $6C_6H_5$), 1.29 (12H, s, $6CH_2Sn$), 1.26 (36H, s, $12CH_3$).

Crystal data

$[Sn_2(C_{10}H_{13})_6(C_8F_4O_4)]$	D_x = 1.344 Mg m^{-3}
M_r = 1272.68	Mo $K\alpha$ radiation
Monoclinic, *C*2	Cell parameters from 8235 reflections
a = 14.4124 (6) Å	θ = 2.2–24.4°
b = 12.5830 (6) Å	μ = 0.85 mm^{-1}
c = 17.9361 (8) Å	T = 295 (2) K
β = 104.833 (1)°	Plate, colorless
V = 3144.3 (2) Å^3	0.18 × 0.15 × 0.08 mm
Z = 2	

Data collection

Bruker SMART APEX area-detector diffractometer	7137 independent reflections
φ and ω scans	6453 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)	R_{int} = 0.030
T_{min} = 0.759, T_{max} = 0.935	θ_{max} = 27.5°
18250 measured reflections	$h = -18 \rightarrow 18$
	$k = -16 \rightarrow 16$
	$l = -23 \rightarrow 23$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0386P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.028	$(\Delta/\sigma)_{max}$ = 0.003
$wR(F^2)$ = 0.068	$\Delta\rho_{max}$ = 0.58 e Å^{-3}
S = 0.99	$\Delta\rho_{min}$ = −0.23 e Å^{-3}
7137 reflections	Absolute structure: Flack (1983), 3565 Friedel pairs
358 parameters	Flack parameter = −0.03 (2)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Sn1—O1	2.085 (2)	Sn1—C11	2.149 (3)
Sn1—C1	2.151 (3)	Sn1—C21	2.155 (4)
O1—Sn1—C1	103.5 (2)	C1—Sn1—C11	117.3 (1)
O1—Sn1—C11	91.9 (1)	C1—Sn1—C21	118.0 (2)
O1—Sn1—C21	104.2 (1)	C11—Sn1—C21	116.0 (1)

H atoms were placed at calculated positions [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] and were included in the refinement in the riding-model approximation.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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