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μ-Phthalato-bis[tris(2-methyl-2-phenylpropyl)tin(IV)]

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Key indicators

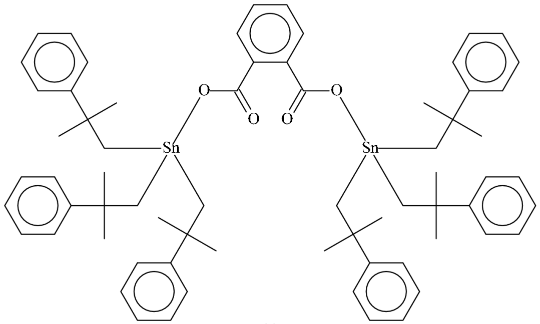
Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.007 Å
R factor = 0.055
wR factor = 0.115
Data-to-parameter ratio = 18.5

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

The title compound, $[Sn_2(C_{10}H_{13})_6(C_8H_4O_4)]$, crystallizes with one molecule per asymmetric unit. The phthalate dianion binds two sterically crowded triorganotin entities and both Sn atoms exist in tetrahedral environments.

Comment

As recently found, the molecule of bis[tris(2-methyl-2-phenylpropyl)tin] tetrafluorophthalate lies on a twofold axis that relates one R_3Sn entity to the other (Tian *et al.*, 2004). In the title unsubstituted phthalate, (I), all atoms lie in general positions, and both Sn atoms are four-coordinate in tetrahedral environments (Fig. 1). Bond dimensions, such as the covalent Sn—O distances, are similar to those found in the fluoro-substituted carboxylate. The less bulky bis(triphenyltin) phthalate analogue also has its metal atom in a tetrahedral geometry (James *et al.*, 1998).



(I)

Experimental

The title compound, (I), was synthesized by condensing bis[tri(2-phenyl-2-methylpropyl)tin] oxide (2.11 g, 2 mmol) with an excess of phthalic acid (0.33 g, 2 mmol) in benzene (60 ml). Water was removed with a Dean–Stark water separator, and the condensation was complete in about 6 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 70% yield; m.p. 405–406 K. Analysis found: C 67.78, H 6.65%; calculated for $C_{68}H_{82}O_4Sn_2$: C 68.02, H 6.88%. IR (KBr disc): ν_{as}(COO) 1659, ν_s(COO) 1348 cm^{-1}.

Crystal data

$[Sn_2(C_{10}H_{13})_6(C_8H_4O_4)]$
M_r = 1200.72
Monoclinic, $P2_1/n$
a = 9.5514 (4) Å
b = 18.0919 (8) Å
c = 35.497 (2) Å
β = 95.170 (1)°
V = 6109.0 (5) Å^3
Z = 4

D_x = 1.306 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5502 reflections
θ = 2.3–20.0°
μ = 0.86 mm^{-1}
T = 295 (2) K
Needle, colorless
0.17 × 0.06 × 0.05 mm

doi:10.1107/S1600536804031952

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{\min} = 0.579$, $T_{\max} = 0.958$
43 944 measured reflections
10 778 independent reflections
7651 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.073$
$\theta_{\max} = 25.0°$
$h = -11 \rightarrow 11$
$k = -21 \rightarrow 21$
$l = -42 \rightarrow 42$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.115$
$S = 1.02$
10 778 reflections
583 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.58$ e Å^{-3}
$\Delta\rho_{\min} = -0.46$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—O1	2.071 (3)	Sn2—O3	2.081 (3)
Sn1—C1	2.143 (5)	Sn2—C31	2.138 (5)
Sn1—C11	2.139 (5)	Sn2—C41	2.151 (5)
Sn1—C21	2.147 (5)	Sn2—C51	2.148 (5)
O1—Sn1—C1	106.8 (2)	O3—Sn2—C31	104.1 (2)
O1—Sn1—C11	101.1 (2)	O3—Sn2—C41	106.3 (2)
O1—Sn1—C21	93.4 (2)	O3—Sn2—C51	93.7 (2)
C1—Sn1—C11	119.4 (2)	C31—Sn2—C41	119.4 (2)
C1—Sn1—C21	116.7 (2)	C31—Sn2—C51	115.0 (2)
C11—Sn1—C21	113.9 (2)	C41—Sn2—C51	114.0 (2)

Phenyl rings were refined as rigid hexagons. H atoms were placed in calculated positions [C—H = 0.93 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ for the phenyl H atoms, C—H = 0.96 Å and $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$ for the methyl H atoms, and C—H = 0.97 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ for the 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.

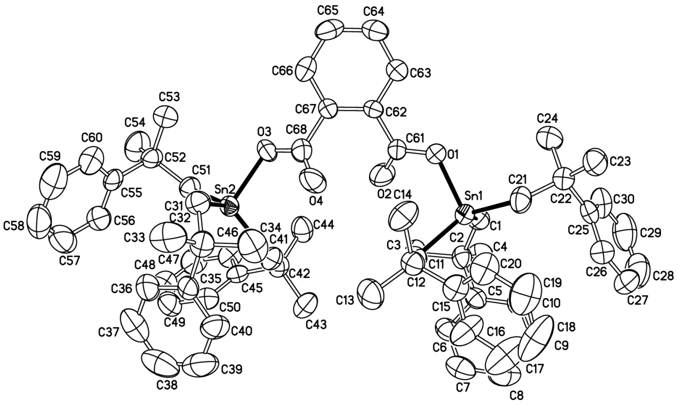


Figure 1
*ORTEP*II (Johnson, 1976) plot of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted.

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References

Bruker (2002). *SADABS*, *SAINT* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
James, B. D., Kivlighon, L. M., Skelton, B. W. & White, A. H. (1998). *Appl. Organomet. Chem.* **12**, 13–23.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Tian, L.-J., Sun, Y.-X. & Ng, S. W. (2004). *Acta Cryst.* E**60**, m1752–m1753.