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Tris(2-methyl-2-phenylpropyl)(pyridine-3-carboxylato)tin(IV)

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The Sn atom of the title compound, $[Sn(C_{10}H_{13})_3(C_6H_4NO_2)]$, is four-coordinate and possesses a distorted tetrahedral geometry.

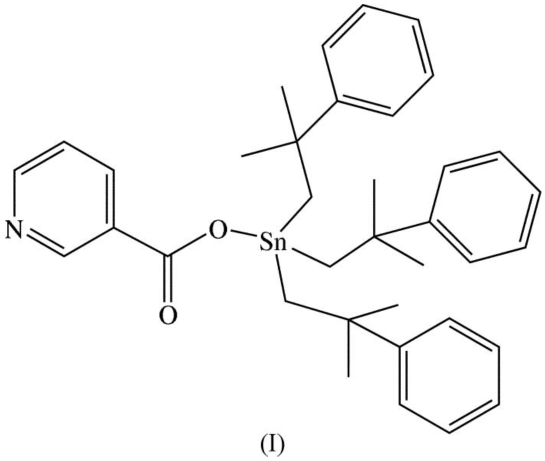
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.030
wR factor = 0.079
Data-to-parameter ratio = 19.8

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

Comment

In several tris(2-methyl-2-phenylpropyl)tin carboxylates, such as tris(2-methyl-2-phenylpropyl)tin phenoxyacetate (Bao *et al.*, 1998), acetate (Bomfim *et al.*, 2002), arylcarboxylate with an organogermyl substituent (Fang *et al.*, 2001) and bis[tris(2-methyl-2-phenylpropyl)tin(IV)] phthalate (Tian *et al.*, 2005), the Sn atoms possess a tetrahedral geometry and do not associate into chain structures *via* carboxylate bridging, due to the effects of the bulky organic groups. In the title compound, (I), tetrahedral coordination is also observed (Fig. 1).



(I)

The Sn···O2 separation of 3.032 (1) Å in (I) indicates that there is a weak interaction between these atoms, which distorts the tetrahedral geometry by opening up the C7—Sn1—C27 angle to 119.94 (9)° and reducing the O1—Sn1—C17 angle to 93.64 (8)°. The bond dimensions around the Sn atom in (I) (Table 1) are similar to those found in the carboxylate structures mentioned above.

There are no intermolecular Sn···N interactions in the crystal structure of (I); such interactions are found in triphenyltin 3-pyridinecarboxylate (Ng *et al.*, 1989), tribenzyltin 3-pyridinecarboxylate (Yin *et al.*, 2003) and tri(2-fluorobenzyl)tin 3-pyridinecarboxylate (Yin *et al.*, 2004).

Experimental

The title compound was synthesized by condensing bis[tri(2-phenyl-2-methylpropyl)tin] oxide (1.05 g, 1 mmol) with 3-pyridinecarboxylic

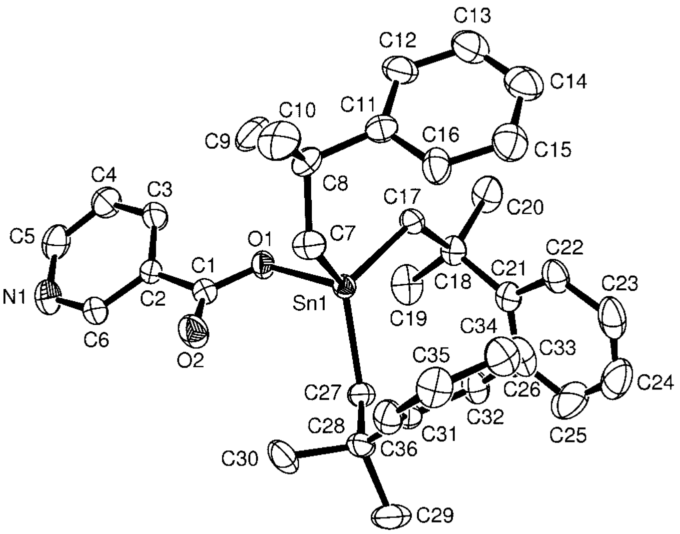


Figure 1

The structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted. For phenyl group C11–C16, the minor disordered component has been omitted for clarity.

acid (0.25 g, 2 mmol) in benzene (50 ml). Water was removed with a Dean–Stark trap and the condensation was complete in about 5 h. The resulting clear solution was evaporated under vacuum. The white solid obtained, (I), was recrystallized from ethanol and crystals of (I) were obtained from cyclohexane by slow evaporation at 298 K (yield 73.5%, m.p. 348–349 K). Analysis, found: C 67.62, H 6.66, N 2.17%; calculated for $C_{36}H_{43}NO_2Sn$: C 67.51, H 6.77, N 2.19%. Spectroscopic analysis: IR (KBr disc): ν_{as}(COO) 1651, ν_s(COO) 1339 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$, δ, p.p.m.): 9.10, 8.80, 8.21, 7.48 (4H, pyridine-H), 7.30–7.12 (15H, *m*, 3 C_6H_5), 1.29 (6H, *s*, 3 CH_2Sn), 1.24 (18H, *s*, 6 CH_3); ^{119}Sn NMR (111.9 MHz, $CDCl_3$, δ, p.p.m.): 104.7.

Crystal data

$[Sn(C_{10}H_{13})_3(C_6H_4NO_2)]$
$M_r = 640.40$
Monoclinic, $P2_1/n$
$a = 12.0660$ (5) Å
$b = 18.2275$ (8) Å
$c = 15.1284$ (6) Å
$\beta = 93.774$ (1)°
$V = 3320.0$ (2) Å^3
$Z = 4$

$D_x = 1.281$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7633 reflections
$\theta = 2.2$–28.3°
$\mu = 0.80$ mm^{-1}
$T = 295$ (2) K
Block, colourless
0.20 × 0.18 × 0.15 mm

Data collection

Bruker SMART APEX detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.856$, $T_{max} = 0.889$
37665 measured reflections

7571 independent reflections
6225 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 27.5°$
$h = -15 \rightarrow 15$
$k = -23 \rightarrow 23$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.080$
$S = 1.03$
7571 reflections
383 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0439P)^2 + 0.4744P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—O1	2.0836 (15)	Sn1—C27	2.142 (2)
Sn1—C17	2.141 (2)	Sn1—C7	2.143 (2)
O1—Sn1—C17	93.64 (8)	O1—Sn1—C7	103.02 (9)
O1—Sn1—C27	104.72 (8)	C17—Sn1—C7	114.26 (9)
C17—Sn1—C27	115.80 (9)	C27—Sn1—C7	119.94 (9)

One phenyl group (C11–C16) is disordered over two positions; *ipso* atom C11 was refined with full occupancy, while the other atoms were refined in two parts, with site occupancy factors of 0.585 (8) (atoms C12–C16) and 0.415 (8) (atoms C12′–C16′). The phenyl rings were restrained to be planar regular hexagons, with target C$\cdots$C distances of 1.38 (1) Å. H atoms were placed in 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: *ORTEP3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Acta Cryst. (2005). E**61**, m1346–m1347 Tian *et al.* • $[Sn(C_{10}H_{13})_3(C_6H_4NO_2)]$ **m1347**