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(μ_3-1,3,5-Benzenetricarboxylato)tris[triphenyltin(IV)] trichloromethane solvate

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Key indicators

Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.014 Å
R factor = 0.064
wR factor = 0.139
Data-to-parameter ratio = 17.6

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

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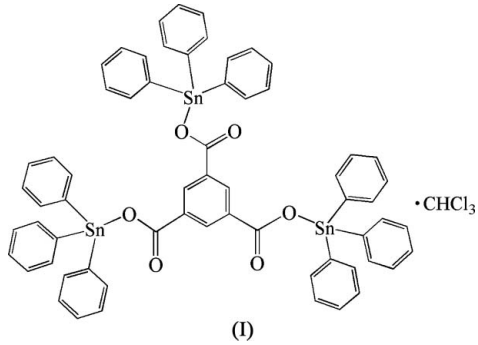
In the title compound, $[Sn_3(C_6H_5)_9(C_9H_3O_6)]\cdot CHCl_3$, the 1,3,5-benzenetricarboxylate trianion binds to three triphenyltin entities and the three Sn atoms exist in distorted tetrahedral $SnOC_3$ environments.

Comment

Recently, (1,3,5-benzenetricarboxylato)tris[triphenyltin(IV)] was prepared by the reaction of 1,3,5-benzenetricarboxylic acid and sodium ethoxide with triphenyltin chloride in a 1:3:3 stoichiometry and the structure of its dichloromethane solvate showed that the Sn atoms possess a distorted tetrahedral geometry (Ma *et al.*, 2005). In the related title compound, (I), the three Sn atoms are also four-coordinate in distorted tetrahedral environments (Fig. 1), with bond angles around the Sn atoms lying in the range 94.6 (2)–119.0 (3)° (Table 1). The C—O distances of the three carboxylate groups indicate that the bonding is essentially localized.



(I)

The Sn1···O2, Sn2···O4 and Sn3···O6 separations are 2.736 (4), 2.793 (4) and 2.751 (5) Å, respectively, indicating there are weak interactions between these atoms, which distort the tetrahedral $SnOC_3$ polyhedra by opening up the angles C10—Sn1—C22, C34—Sn2—C40, and C52—Sn3—C58 and reducing O1—Sn1—C16, O3—Sn2—C28, and O5—Sn3—C46. In general, the geometries around the Sn atoms, such as the covalent Sn—O distances, are similar to those found in the structures of (1,3,5-benzenetricarboxylato)tris[triphenyltin(IV)] dichloromethane solvate (Ma *et al.*, 2005) and other triphenyltin carboxylates (Tiekink, 1991, 1994).

Experimental

The title compound was synthesized by condensing triphenyltin hydroxide (1.10 g, 3 mmol) with 1,3,5-benzenetricarboxylic acid (0.21 g, 1 mmol) in benzene (50 ml). Water was removed with a Dean–Stark water separator and the condensation was complete in

about 4 h. The resulting clear solution was evaporated under vacuum. The white solid obtained was recrystallized from ethanol and crystals of (I) were obtained from trichloromethane by slow evaporation at 298 K (yield 83.5%, m.p. 389–391 K). Analysis found: C 55.62, H 3.37%; calculated for $C_{64}H_{49}Cl_3O_6Sn_3$: C 55.84, H 3.59%.

Crystal data

$[Sn_3(C_6H_5)_9(C_9H_3O_6)]\cdot CHCl_3$	$V = 2955.1\ (18)$ Å^3
$M_r = 1376.45$	$Z = 2$
Triclinic, $P\overline{1}$	$D_x = 1.547$ Mg m^{-3}
$a = 13.092\ (4)$ Å	Mo $K\alpha$ radiation
$b = 13.990\ (5)$ Å	$\mu = 1.44$ mm^{-1}
$c = 18.207\ (8)$ Å	$T = 291\ (2)$ K
$\alpha = 112.456\ (5)^\circ$	Block, colorless
$\beta = 104.944\ (8)^\circ$	0.12 × 0.10 × 0.06 mm
$\gamma = 90.641\ (6)^\circ$	

Data collection

Bruker SMART APEX CCD diffractometer	24159 measured reflections
	12063 independent reflections
φ and ω scans	6615 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)	$R_{int} = 0.067$
	$\theta_{max} = 26.5^\circ$
$T_{min} = 0.846$, $T_{max} = 0.918$	

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.064$	$w = 1/[\sigma^2(F_o^2) + (0.0333P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.139$	$(\Delta/\sigma)_{max} = 0.001$
$S = 0.96$	$\Delta\rho_{max} = 0.65$ e Å^{-3}
12063 reflections	$\Delta\rho_{min} = -0.53$ e Å^{-3}
685 parameters	

Table 1
Selected geometric parameters (Å, °).

Sn1—O1	2.067 (4)	Sn3—C52	2.104 (8)
Sn1—C16	2.101 (7)	Sn3—C58	2.116 (8)
Sn1—C10	2.106 (8)	Sn3—C46	2.119 (7)
Sn1—C22	2.128 (7)	C1—O1	1.289 (8)
Sn2—O3	2.060 (4)	C1—O2	1.215 (7)
Sn2—C40	2.112 (7)	C8—O3	1.297 (8)
Sn2—C34	2.122 (8)	C8—O4	1.208 (8)
Sn2—C28	2.131 (7)	C9—O5	1.295 (8)
Sn3—O5	2.066 (4)	C9—O6	1.239 (8)
O1—Sn1—C16	95.7 (2)	O3—Sn2—C28	94.6 (2)
O1—Sn1—C10	112.0 (2)	C40—Sn2—C28	111.9 (3)
C16—Sn1—C10	108.8 (3)	C34—Sn2—C28	110.5 (3)
O1—Sn1—C22	110.0 (2)	O5—Sn3—C52	108.8 (2)
C16—Sn1—C22	111.7 (3)	O5—Sn3—C58	111.6 (2)
C10—Sn1—C22	116.6 (3)	C52—Sn3—C58	116.7 (3)
O3—Sn2—C40	108.6 (2)	O5—Sn3—C46	96.0 (2)
O3—Sn2—C34	109.4 (2)	C52—Sn3—C46	112.1 (3)
C40—Sn2—C34	119.0 (3)	C58—Sn3—C46	109.7 (3)

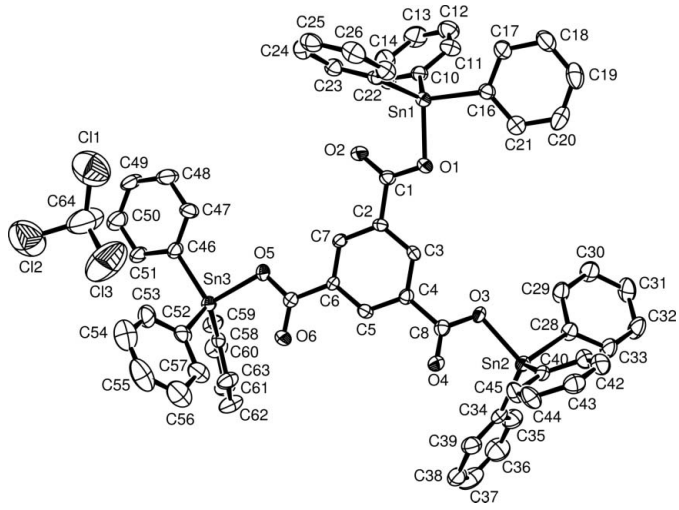


Figure 1
The structure of (I), showing 30% probability displacement ellipsoids. H atoms have been omitted for clarity.

The C···C distances of one phenyl ring (C46–C51) were restrained to be 1.38 (1) Å. H atoms were placed at calculated positions (C—H = 0.93–0.98 Å) and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$.

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: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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