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Di-μ_2-chloro-1:2κ^2*Cl*,3:4κ^2*Cl*-chloro-4κ*Cl*-(6-chloropyridine-3-carboxylato-1κ^2*O*,*O*′)octamethyl-1κ^2*C*,2κ^2*C*,3κ^2*C*,4κ^2*C*-di-μ_3-oxo-1:2:3κ^3*O*,2:3:4κ^3*O*-tetratin(IV) diethyl ether solvate

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In the title compound, $[Sn_4(CH_3)_8(C_6H_3ClNO_2)Cl_3O_2]\cdot C_4H_{10}O$, Cl and O atoms bridge the Sn species, resulting in a cluster containing four metal atoms. Three of the four Sn coordination polyhedra are trigonal bipyramids; the other is irregular six-coordinate. Long Sn$\cdots$O and Sn$\cdots$N interactions result in the formation of a sheet structure.

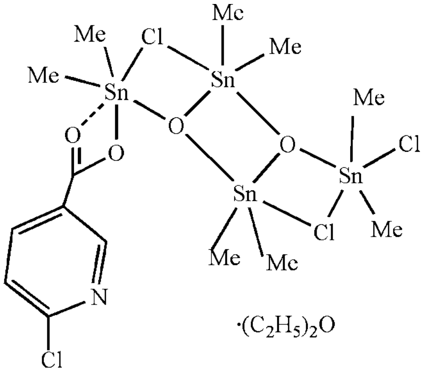
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.008 Å
Some non-H atoms missing
R factor = 0.032
wR factor = 0.089
Data-to-parameter ratio = 25.9

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

Comment

The main molecule of the title compound, (I) (Fig. 1), contains four Sn atoms assembled about a central Sn_2O_2 core. The three-coordinate bridging atoms O1 and O2 in the Sn_2O_2 ring are also attached to a terminal Me_2Sn unit; a chloride ion also bridges to this terminal grouping.



(I)

The geometries of Sn2, Sn3 and Sn4 are distorted trigonal–bipyramidal (Table 1). Atoms Sn2, O1 and Cl13 are in axial positions [O1—Sn2—Cl3 = 153.12 (15)°], and the C atoms of the two methyl groups and O2 are in equatorial positions. The sum of the equatorial C—Sn—C and O—Sn—C angles is 359.5°, indicating approximate coplanarity for these atoms.

The geometry of Sn1 is irregular; the C1 carboxylate group coordinates very asymmetrically resulting in a very long Sn1—O4 bond. The C1—O3 and C1—O4 bond lengths suggest that the carboxylate charge is essentially localized (Table 1).

This unusual Sn1 coordination may arise because of supramolecular interactions in the crystal structure. An Sn1$\cdots$O4($2 - x$, $1 - y$, $1 - z$) contact of 3.182 (6) Å occurs, roughly bisecting the C7—Sn1—C8 grouping, leading to dimeric associations of $C_{16}H_{32}Cl_4NO_{4.5}Sn_4$ molecules *via* a second Sn_2O_2 unit (Fig. 2).

If a long Sn4$\cdots$N1($1 - x$, $1 - y$, $1 - z$) contact of 3.443 (1) Å is considered to be a bonding interaction (van der Waals radius sum = 3.81 Å), then a two-dimensional network is formed (Fig. 2). The local structure of (I) is similar to that seen previously in a related compound (Reyes *et al.*, 2003).

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Experimental

The reaction was carried out under a nitrogen atmosphere. 6-Chloronicotinic acid (0.1576 g, 1 mmol) and sodium ethoxide (0.0681 g, 1 mmol) were added to benzene (80 ml) in a Schlenk flask, and the mixture was stirred for 20 min; Me_2SnCl_2 (0.4394 g, 2 mmol) was then added to the mixture, which was stirred for 12 h at 323 K. After cooling to room temperature, the solution was filtered. The solvent of the filtrate was gradually removed by evaporation under vacuum until a solid product was obtained. The solid was then recrystallized from diethyl ether to yield blocks of (I) (yield 0.6489 g, 70%; m.p. 411 K).

Crystal data

$[Sn_4(CH_3)_8(C_6H_3ClNO_2)Cl_3O_2]\cdot C_4H_{10}O$
$M_r = 964.14$
Monoclinic, $P2_1/n$
$a = 16.123$ (2) Å
$b = 11.0953$ (15) Å
$c = 20.309$ (3) Å
$\beta = 113.059$ (2)°
$V = 3342.7$ (8) Å^3
$Z = 4$
$D_x = 1.939$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 3.30$ mm^{-1}
$T = 298$ (2) K
Block, colorless
0.42 × 0.29 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.338$, $T_{max} = 0.588$ (expected range = 0.317–0.552)
17200 measured reflections
6521 independent reflections
4331 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 26.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.089$
$S = 1.08$
6521 reflections
252 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.04P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.57$ e Å^{-3}
$\Delta\rho_{min} = -0.79$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—O1	2.031 (3)	Sn3—C12	2.083 (6)
Sn1—C8	2.091 (5)	Sn3—C11	2.092 (6)
Sn1—C7	2.096 (5)	Sn3—O2	2.134 (3)
Sn1—O3	2.148 (3)	Sn3—Cl2	2.6434 (15)
Sn1—O4	2.690 (4)	Sn4—O2	2.023 (3)
Sn1—Cl2	2.9098 (15)	Sn4—C13	2.094 (6)
Sn2—O2	2.038 (3)	Sn4—C14	2.096 (6)
Sn2—C9	2.087 (5)	Sn4—Cl4	2.4649 (17)
Sn2—C10	2.108 (5)	Sn4—Cl3	2.7730 (15)
Sn2—O1	2.122 (3)	C1—O4	1.226 (6)
Sn2—Cl3	2.6701 (15)	C1—O3	1.296 (6)
Sn3—O1	2.032 (3)		
O1—Sn1—C8	105.51 (18)	O3—Sn1—O4	52.46 (11)
O1—Sn1—C7	104.75 (19)	O1—Sn1—Cl2	73.60 (9)
C8—Sn1—C7	145.4 (2)	C8—Sn1—Cl2	85.00 (17)
O1—Sn1—O3	82.20 (12)	C7—Sn1—Cl2	87.68 (16)
C8—Sn1—O3	100.49 (19)	O3—Sn1—Cl2	155.76 (9)
C7—Sn1—O3	99.95 (18)	O4—Sn1—Cl2	151.68 (8)
O1—Sn1—O4	134.66 (11)	C1—Sn1—Cl2	176.37 (11)
C8—Sn1—O4	84.63 (18)	C1—O3—Sn1	105.2 (3)
C7—Sn1—O4	86.03 (18)	C1—O4—Sn1	81.4 (3)

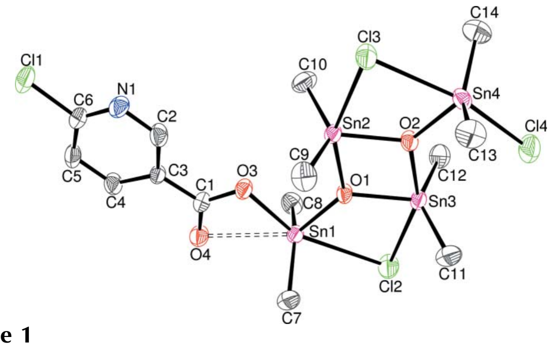

Figure 1
The molecular structure of (I) with 30% displacement ellipsoids (H atoms omitted for clarity).

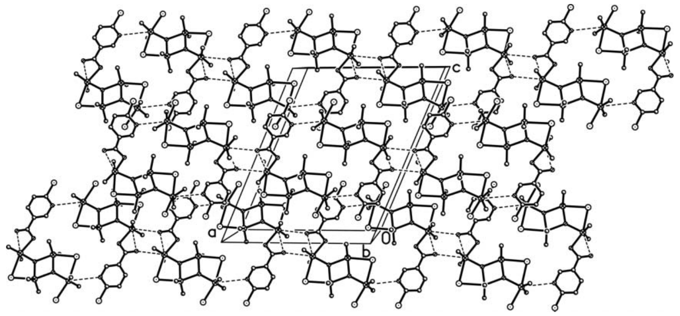

Figure 2
View down [010] of a section of the two-dimensional supramolecular network in the crystal structure of (I), showing the intermolecular Sn···O and Sn···N interactions as dashed lines. H atoms have been omitted.

The main molecule is accompanied by a highly mobile/disordered diethyl ether molecule. Attempts to model this species were not successful, so the SQUEEZE option of *PLATON* (Spek, 2003) was used to remove the contribution of this species. The site occupancies of the ether atoms are not certain; full occupancy was assumed for the determination of overall molecular weight *etc*. H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(methyl C).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); 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, 1998)*; software used to prepare material for publication: *SHELXL97*.

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