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Di-μ_2-chloro-dichlorooctakis(2-fluorobenzyl)di-μ_3-oxo-tetratin(IV) dihydrate

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In the title compound, $[Sn_4(C_7H_6F)_8Cl_4O_2]\cdot 2H_2O$, the Cl and O atoms bridge four Sn atoms, forming a tetranuclear complex which is located on an inversion centre. The four Sn atoms are coplanar with the bridging Cl and O atoms, and the complex shows a ladder structure. Each Sn atom assumes a distorted trigonal–bipyramidal geometry.

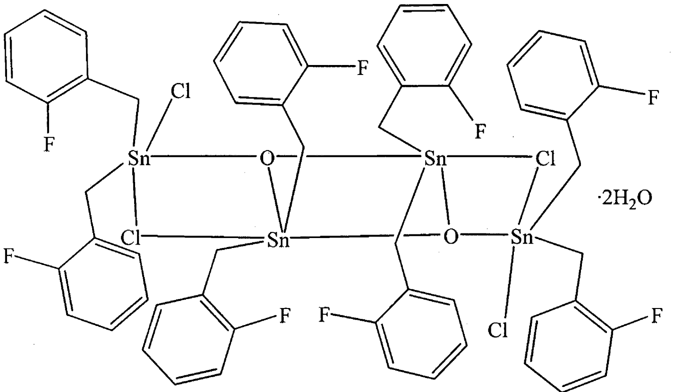
Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.009 Å
Disorder in main residue
R factor = 0.027
wR factor = 0.071
Data-to-parameter ratio = 13.2

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

Comment

Partial hydrolysis of diorganotin dihalides leads to interesting classes of tetraorganodistannoxanes, $[R_2X\text{SnOSn}R_2X]_2$ and $[R_2X\text{SnOSn}R_2\text{O}]_2$. Different types of geometry, ladder and staircase, for these two kinds of complexes have been reported (Hunter & Sanders, 1990). As part of an investigation into the relationship between the structure and hydrolysis course, we present here the structure of the title Sn^{IV} compound, (I).



(I)

The structure of (I) comprises the tetranuclear Sn^{IV} complex and uncoordinated water molecules. The Sn^{IV} complex is located on an inversion centre (Fig. 1). The asymmetric unit contains two Sn atoms with different coordination environments, both having a distorted trigonal–bipyramidal geometry. While atom Sn1 is coordinated by two Cl, one O and two fluorobenzyl ligands, Sn2 is surrounded by one Cl, two O and two fluorobenzyl ligands. Atom Cl1 is terminally coordinated to atom Sn1 with a shorter Sn—Cl bond distance, whereas atom Cl2 plays the role of a bridging ligand, with rather longer Sn—Cl bond distances (Table 1). The O atoms each bridge three Sn atoms, forming the tetranuclear complex.

The four Sn atoms are coplanar with the bridging Cl and O atoms, the maximum atomic deviation being 0.0054 (3) Å. The complex thus displays a ladder structure, which is different from the staircase structure for the related Sn^{IV} complex $[Me_4Sn_2(OSiMe_3)_2O]_2$ (Okawara & Wada, 1963), but similar

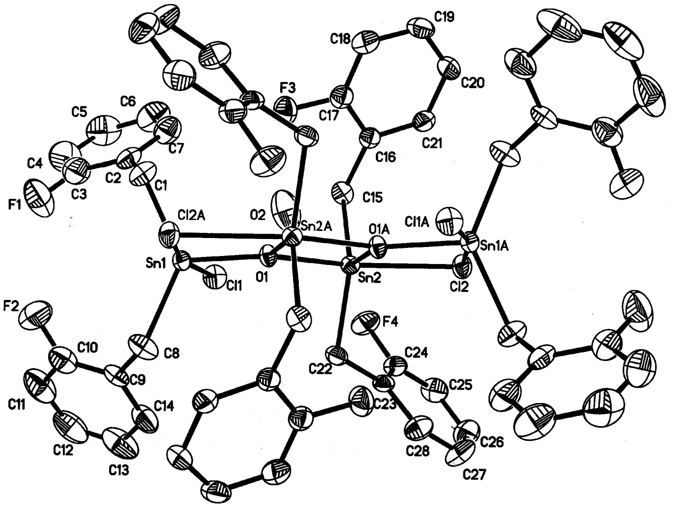


Figure 1
The molecular structure of (I), with 30% probability displacement ellipsoids [symmetry code: (A) $1 - x$, $1 - y$, $1 - z$]. H atoms and the minor disorder components have been omitted for clarity.

to other chlorodistannoxanes reported previously (Dokorou *et al.*, 2001).

Experimental

$(2\text{-}FC_6H_4CH_2)_2SnCl_2$ (2 mmol) was dissolved in an ethanol solution (30 ml, 95%) of NaOH (2 mmol). The solution was refluxed for 5 h and the solvent was then removed by evaporation *in vacuo*. The crude adduct was separated and recrystallized from dichloromethane. Single crystals of (I) were obtained after several days (m.p. 483 K).

Crystal data

$[Sn_4(C_7H_6F)_8Cl_4O_2]\cdot 2H_2O$
$M_r = 1557.54$
Triclinic, $P\overline{1}$
$a = 10.6243$ (17) Å
$b = 10.8750$ (18) Å
$c = 14.157$ (2) Å
$\alpha = 104.983$ (2)°
$\beta = 107.084$ (2)°
$\gamma = 91.115$ (2)°
$V = 1502.3$ (4) Å^3

$Z = 1$
$D_x = 1.722$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5183 reflections
$\theta = 2.7$–25.0°
$\mu = 1.89$ mm^{-1}
$T = 273$ (2) K
Block, colourless
0.41 × 0.37 × 0.32 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.480$, $T_{\max} = 0.546$
7857 measured reflections

5182 independent reflections
4257 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.014$
$\theta_{\max} = 25.0°$
$h = -12 \rightarrow 12$
$k = -8 \rightarrow 12$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.071$
$S = 1.07$
5182 reflections
393 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0324P)^2 + 1.4097P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.62$ e Å^{-3}
$\Delta\rho_{\min} = -0.38$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—O1	2.023 (2)	Sn2—O1^{i}	2.049 (2)
Sn1—C8	2.135 (5)	Sn2—C22	2.144 (4)
Sn1—C1	2.146 (4)	Sn2—C15	2.151 (4)
Sn1—Cl1	2.4071 (12)	Sn2—O1	2.162 (2)
Sn1—Cl2^{i}	2.8152 (11)	Sn2—Cl2	2.5713 (11)
O1—Sn1—C8	109.63 (17)	C22—Sn2—C15	127.58 (17)
O1—Sn1—C1	111.86 (16)	O1^{i}—Sn2—O1	73.92 (11)
C8—Sn1—C1	132.0 (2)	C22—Sn2—O1	94.98 (13)
O1—Sn1—Cl1	91.90 (8)	C15—Sn2—O1	100.59 (14)
C8—Sn1—Cl1	101.02 (14)	O1^{i}—Sn2—Cl2	80.21 (7)
C1—Sn1—Cl1	100.62 (13)	C22—Sn2—Cl2	95.83 (12)
O1—Sn1—Cl2^{i}	74.74 (7)	C15—Sn2—Cl2	91.25 (12)
C8—Sn1—Cl2^{i}	85.12 (13)	O1—Sn2—Cl2	154.13 (7)
C1—Sn1—Cl2^{i}	83.51 (13)	Sn2—Cl2—Sn1^{i}	82.79 (3)
Cl1—Sn1—Cl2^{i}	166.57 (4)	Sn1—O1—Sn2^{i}	122.25 (12)
O1^{i}—Sn2—C22	115.25 (15)	Sn1—O1—Sn2	131.65 (12)
O1^{i}—Sn2—C15	117.14 (14)	Sn2^{i}—O1—Sn2	106.08 (10)

Symmetry code: (i) $-x + 1$, $-y + 1$, $-z + 1$.

The uncoordinated water O atom is disordered over two positions; occupancies were initially refined and finally fixed at 0.54 and 0.46. All fluorobenzene groups are disordered over two positions each, with different orientations; all F atoms therefore appeared in both *ortho*-positions with different site occupancy factors. The occupancies were initially refined independently and converged to 0.717 (11) and 0.283 (11) (F1 and F1′), 0.813 (10) and 0.187 (10) (F2 and F2′), 0.914 (8) and 0.086 (8) (F3 and F3′) and 0.912 (9) and 0.088 (9) (F4 and F4′); in the final stage of the refinement, they were fixed at 0.72 and 0.28 (F1 and F1′), 0.81 and 0.19 (F2 and F2′), 0.91 and 0.09 (F3 and F3′) and 0.91 and 0.09 (F4 and F4′). H atoms bonded to C atoms were placed in calculated positions and treated as riding on their parent atoms, with C—H distances of 0.93 Å (aromatic) and 0.97 Å (methylene). Water H atoms were located in a difference Fourier map and refined as riding in their as-found relative positions, with $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(carrier).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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