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Dichlorooctakis(di-2,4-chlorobenzyl)-di-μ_2-methanolato-di-μ_3-oxo-tetratin(IV)

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Key indicators

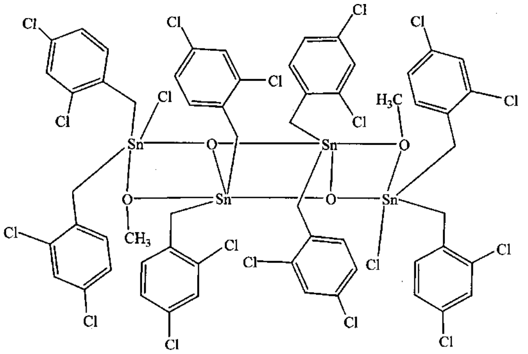
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.014 Å
R factor = 0.045
wR factor = 0.129
Data-to-parameter ratio = 15.8

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

In the title complex, $[Sn_4(C_7H_5Cl_2)_8(CH_3O)_2Cl_2O_2]$, the O atoms and the methoxy groups bridge four Sn atoms to form a tetranuclear complex which is located on an inversion centre. Each Sn atom assumes a distorted trigonal–bipyramidal geometry.

Comment

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



(I)

From Fig. 1, it can be seen that complex (I) has a dimeric formulation and contains two independent pentacoordinated Sn atoms. It can be viewed as a centrosymmetric dimer, where one half of the molecule comprises the crystallographic asymmetric unit and the other half is generated by an inversion centre.

The Sn atoms are pentacoordinated with distorted trigonal–bipyramidal geometries, having two benzyl groups and atom O1 in equatorial positions. The complex molecules have ladder structures rather than staircase arrangements, as evidenced by the near coplanarity of the atoms comprising the fused ring systems. Atoms Sn1, Sn2, O1, O2, Sn1^i, Sn2^i, O1^i and O2^i [symmetry code: (i) $-x + 1, -y + 1, -z + 1$] are coplanar to within 0.032 (3) Å. This near coplanarity is also seen in the dihedral angle between the planes defined by the central and terminal four-atom rings. The dihedral angle between the least-squares mean planes defined by atoms Sn1, O1, Sn2 and O2 [coplanar to within 0.033 (4) Å] and atoms Sn2, O1, Sn2^i and O1^i (required by symmetry to be parallel) is 3.08 (8)°. The ladder structure contrasts with the staircase

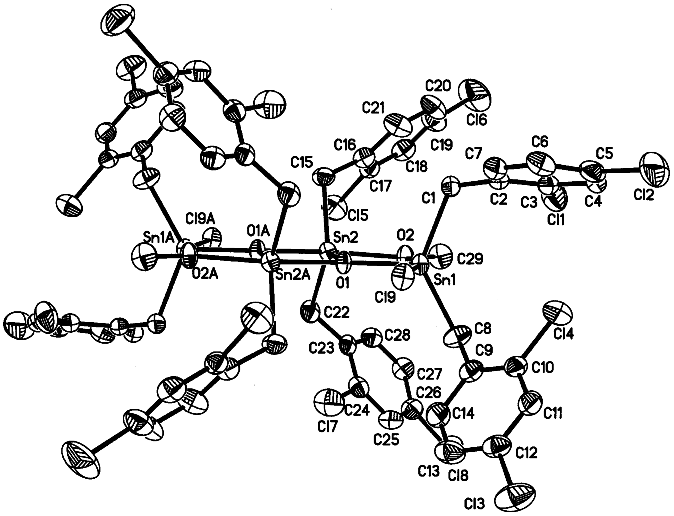


Figure 1
The structure of the title complex, showing 30% probability displacement ellipsoids and the atom-numbering scheme [symmetry code: (A) $-x + 1$, $-y + 1$, $-z + 1$]. H atoms have been omitted for clarity.

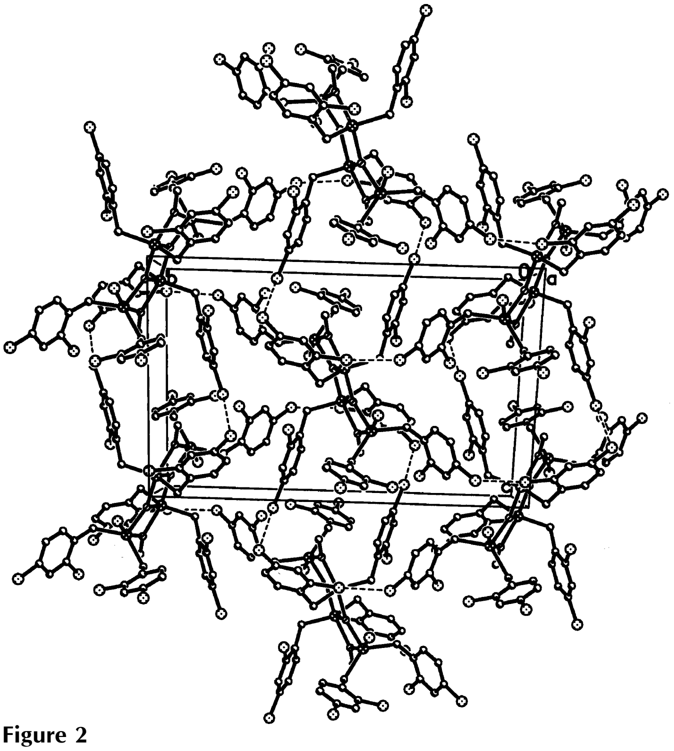


Figure 2
The crystal packing of the title complex.

structure of $[Me_4Sn_2(OSiMe_3)_2O]_2$ (Okawara & Wada, 1963), but it is similar to other chlorodistannoxanes (Dokorou *et al.*, 2001) studied by X-ray diffraction in the appearance of a planar arrangement of the atoms comprising the ladder unit.

Experimental

(Di-2,4-Cl-$C_6H_3CH_2$)$_2$$SnCl_2$ (2 mmol) was added to a solution of sodium methoxide (3 mmol) in methanol (30 ml, 95%). The mixture was then heated under reflux with stirring for 5 h and the solvent was removed by evaporation *in vacuo*. The crude adduct was recrystallized from dichloromethane and colourless crystals suitable for X-ray diffraction were obtained (m.p. 427 K). Analysis calculated for $C_{58}H_{46}Cl_{18}O_4Sn_4$: C 36.29, H 2.42%; found: C 36.20, H 2.45%.

Crystal data

$[Sn_4(C_7H_5Cl_2)_8(CH_3O)_2Cl_2O_2]$
$M_r = 1919.81$
Monoclinic, $P2_1/c$
$a = 12.462$ (3) Å
$b = 21.277$ (5) Å
$c = 14.201$ (3) Å
$\beta = 113.045$ (3)°
$V = 3464.7$ (14) Å^3
$Z = 2$

$D_x = 1.840$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4626 reflections
$\theta = 2.5$–23.8°
$\mu = 2.16$ mm^{-1}
$T = 298$ (2) K
Block, colourless
0.50 × 0.48 × 0.45 mm

Data collection

Siemens SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.351$, $T_{\max} = 0.378$
17614 measured reflections

6006 independent reflections
3838 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.057$
$\theta_{\max} = 25.0°$
$h = -11 \rightarrow 14$
$k = -23 \rightarrow 25$
$l = -16 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.129$
$S = 1.03$
6006 reflections
379 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0483P)^2 + 7.9094P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.92$ e Å^{-3}
$\Delta\rho_{\min} = -0.48$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sn1—O1	2.014 (4)	Sn2—O1	2.040 (4)
Sn1—C8	2.155 (8)	Sn2—O2	2.120 (5)
Sn1—C1	2.164 (8)	Sn2—C22	2.144 (8)
Sn1—O2	2.213 (5)	Sn2—O1$^{\rm i}$	2.145 (4)
Sn1—Cl9	2.442 (2)	Sn2—C15	2.148 (8)
O1—Sn1—C8	116.5 (3)	O2—Sn2—C22	102.6 (3)
O1—Sn1—C1	110.4 (2)	O1—Sn2—O1$^{\rm i}$	73.16 (19)
C8—Sn1—C1	130.7 (4)	O2—Sn2—O1$^{\rm i}$	146.12 (17)
O1—Sn1—O2	71.49 (17)	C22—Sn2—O1$^{\rm i}$	95.1 (3)
C8—Sn1—O2	88.5 (3)	O1—Sn2—C15	113.8 (3)
C1—Sn1—O2	92.4 (3)	O2—Sn2—C15	98.7 (3)
O1—Sn1—Cl9	88.00 (13)	C22—Sn2—C15	125.8 (4)
C8—Sn1—Cl9	98.9 (3)	O1$^{\rm i}$—Sn2—C15	93.8 (3)
C1—Sn1—Cl9	97.1 (2)	Sn1—O1—Sn2	112.9 (2)
O2—Sn1—Cl9	159.36 (13)	Sn1—O1—Sn2$^{\rm i}$	140.0 (2)
O1—Sn2—O2	72.98 (17)	Sn2—O1—Sn2$^{\rm i}$	106.84 (19)
O1—Sn2—C22	119.9 (3)	Sn2—O2—Sn1	102.42 (18)

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

All H atoms were positioned geometrically and refined as riding on their parent atoms, with aromatic C—H distances of 0.93 Å, methyl C—H distances of 0.96 Å and methylene C—H distances of 0.97 Å. The $U_{\rm iso}$(H) values were set at $1.5U_{\rm eq}$(C) for methyl and $1.2U_{\rm eq}$(C) for other C-bound H atoms.

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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