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Di-μ_2-chloro-dichlorodi-μ_3-oxo-octaphenyltetratin dichloromethane solvate

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Key indicators

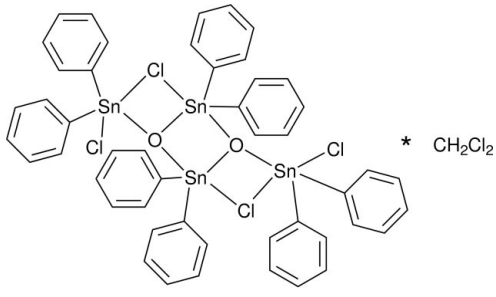
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.008 Å
Disorder in solvent or counterion
R factor = 0.033
wR factor = 0.057
Data-to-parameter ratio = 15.9

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

The title compound [systematic name: 1,2;3,4-di-μ-chloro-1,4-dichloro-1,1,2,2,3,3,4,4-octaphenyl-di-μ_3-oxo-tetratin(IV)], $[Sn_4(C_6H_5)_8Cl_4O_2]$, crystallizes as a CH_2Cl_2 solvate. Unlike the previously known unsolvated form, the structure does not possess a crystallographic inversion center. The dimer consists of a central Sn_2O_2 ring with two additional adjacent Sn_2ClO four-membered rings. The Sn, O and Cl atoms are approximately coplanar; the Sn atoms exhibit a distorted trigonal-bipyramidal configuration.

Comment

Organotin compounds have been the subject of considerable interest in several fields of research in recent years, as a result of the biological properties exhibited by many of these complexes (Arakawa, 1998; Novelli *et al.*, 1999). In addition, organotin complexes have been known to be extremely useful in several catalytic transformations, being used as catalysts, co-catalysts, intermediates or starting materials in important organic transformations (Otera, 1993; Durand *et al.*, 2000; Orita *et al.*, 2001). Moreover, the very rich structural diversity that organotin complexes exhibit remains a continuous field of research for a considerable group of scientists (Holmes, 1989; Beckmann & Jurkschat, 2001).



(I)

The title compound has been prepared previously (Vollano *et al.*, 1984) using acridine as a base and benzene as the reaction and crystallization solvent, and crystallized in an unsolvated form, (II). In the present study, for the preparation of the title compound, (I), triethylamine was used as a base and CH_2Cl_2 as solvent. The crystal data and coordinates of (II) are reported in the Cambridge Structural Database (Version 5.25; refcode CIJJIO; Allen, 2002).

In the structure of (II), molecules are found as centrosymmetric dimers. In contrast, as shown in Fig. 2, the molecules of (I) do not possess a crystallographic inversion center and, to the best of our knowledge, represent the first example

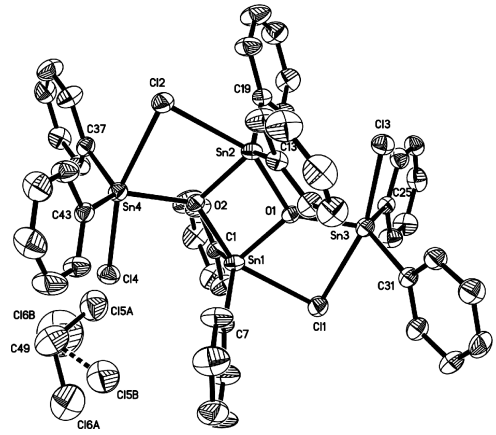


Figure 1
The molecular structure of (I), with displacement ellipsoids at the 30% probability level. Some C-atom labels and all H atoms have been omitted for clarity. The two disorder components of the solvent molecule are shown.

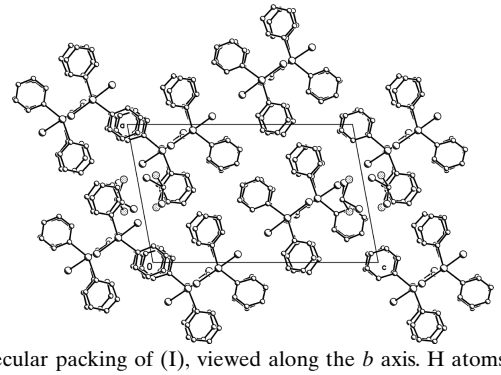


Figure 2
The molecular packing of (I), viewed along the *b* axis. H atoms and the disordered solvent molecules have been omitted for clarity.

among the structures possessing the dimeric distannoxane moiety.

Despite this difference, the geometric parameters in Table 1 correlate very well with those reported by Vollano *et al.* (1984). In fact, the r.m.s. deviation for the Sn, O and Cl framework is 0.048 Å for a least-squares fit of the two structures, the major differences being the orientation of the phenyl ring substituents. From these facts, the 'ladder' structure for the whole molecule and distorted trigonal-bipyramidal coordination polyhedra for the Sn atoms previously observed are confirmed.

Experimental

To a solution of $Ph_2Sn_2Cl_2$(200 mg, 0.5817 mmol) in CH_2Cl_2 (15 ml) was added a solution of triethylamine (147.16 mg, 1.45 mmol) in CH_2Cl_2 (10 ml). The solution was stirred magnetically with gentle warming (313 K) for 48 h. After this period, the solution was filtered through a short plug of celite and the solvent was removed under vacuum. The white residue was crystallized from methanol to afford 141 mg (77% yield) of $[Ph_2(Cl)SnOSnPh_2(Cl)]_2$ as a microcrystalline white powder. An unsolvated sample was used for melting-point determination and chemical analysis. Recrystallization from a double-layer solvent system (CH_2Cl_2/MeOH) afforded colorless crystals suitable for X-ray analysis. M.p. 467–469 K. Analysis found: C 45.77, H 3.11%; $C_{24}H_{20}Cl_2OSn$ requires: C 45.56, H 3.16%. MS–FAB$^+$: M/z 923 [M^+ (Ph_2SnCl_2)].

Crystal data

$[Sn_4(C_6H_5)_8Cl_4O_2]\cdot CH_2Cl_2$	D_x = 1.784 Mg m^{-3}
M_r = 1350.29	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 6333 reflections
a = 11.694 (1) Å	θ = 2.3–25.0°
b = 23.256 (1) Å	μ = 2.32 mm^{-1}
c = 18.805 (1) Å	T = 293 (2) K
β = 100.565 (1)°	Prism, colorless
V = 5027.4 (6) Å^3	0.24 × 0.17 × 0.11 mm
Z = 4	

Data collection

Bruker SMART APEX CCD diffractometer	8864 independent reflections
ω scans	6043 reflections with $I > 2\sigma(I)$
Absorption correction: analytical face-indexed (*XPREP* in *SAINT-Plus*; Bruker, 1999)	R_{int} = 0.072
	θ_{max} = 25.0°
T_{min} = 0.758, T_{max} = 0.878	$h = -13 \rightarrow 13$
40974 measured reflections	$k = -27 \rightarrow 27$
	$l = -22 \rightarrow 22$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.033	$w = 1/[\sigma^2(F_o^2) + (0.0005P)^2]$ where $P = (F_o^2+2F_c^2)/3$
$wR(F^2)$ = 0.057	$(\Delta/\sigma)_{max}$ = 0.012
S = 0.98	$\Delta\rho_{max}$ = 0.57 e Å^{-3}
8864 reflections	$\Delta\rho_{min}$ = −0.50 e Å^{-3}
559 parameters	

Table 1
Selected geometric parameters (Å, °).

Sn1—O1	2.052 (3)	Sn2—O2	2.052 (3)
Sn1—C7	2.096 (4)	Sn2—C13	2.109 (4)
Sn1—C1	2.101 (4)	Sn2—C19	2.114 (4)
Sn1—O2	2.109 (2)	Sn2—O1	2.128 (2)
Sn1—Cl1	2.6881 (12)	Sn2—Cl2	2.6557 (12)
Sn3—O1	2.032 (3)	Sn4—O2	2.026 (3)
Sn3—C25	2.108 (5)	Sn4—C37	2.114 (5)
Sn3—C31	2.121 (5)	Sn4—C43	2.118 (5)
Sn3—Cl3	2.4460 (12)	Sn4—Cl4	2.4397 (12)
Sn3—Cl1	2.6847 (13)	Sn4—Cl2	2.7358 (12)
O1—Sn1—C7	110.32 (15)	C13—Sn2—O1	100.04 (15)
O1—Sn1—C1	112.16 (15)	C19—Sn2—O1	99.14 (14)
C7—Sn1—C1	135.65 (18)	O2—Sn2—Cl2	77.06 (7)
O1—Sn1—O2	74.68 (10)	C13—Sn2—Cl2	90.56 (13)
C7—Sn1—O2	100.92 (14)	C19—Sn2—Cl2	90.78 (12)
C1—Sn1—O2	101.91 (14)	O1—Sn2—Cl2	151.31 (8)
O1—Sn1—Cl1	76.56 (7)	O2—Sn4—C37	114.11 (14)
C7—Sn1—Cl1	89.02 (12)	O2—Sn4—C43	115.76 (15)
C1—Sn1—Cl1	88.82 (12)	C37—Sn4—C43	128.11 (17)
O2—Sn1—Cl1	151.24 (8)	O2—Sn4—Cl4	86.28 (8)
O1—Sn3—C25	116.64 (14)	C37—Sn4—Cl4	98.36 (13)
O1—Sn3—C31	116.02 (14)	C43—Sn4—Cl4	98.20 (13)
C25—Sn3—C31	125.97 (17)	O2—Sn4—Cl2	75.54 (8)
O1—Sn3—Cl3	85.26 (8)	C37—Sn4—Cl2	89.69 (13)
C25—Sn3—Cl3	97.32 (13)	C43—Sn4—Cl2	89.40 (13)
C31—Sn3—Cl3	98.11 (13)	Cl4—Sn4—Cl2	161.82 (4)
O1—Sn3—Cl1	76.94 (8)	Sn3—Cl1—Sn1	83.71 (3)
C25—Sn3—Cl1	90.86 (13)	Sn2—Cl2—Sn4	83.65 (3)
C31—Sn3—Cl1	89.70 (13)	Sn3—O1—Sn1	122.78 (12)
Cl3—Sn3—Cl1	162.21 (4)	Sn3—O1—Sn2	132.02 (14)
O2—Sn2—C13	112.58 (15)	Sn1—O1—Sn2	105.17 (12)
O2—Sn2—C19	109.83 (14)	Sn4—O2—Sn2	123.73 (12)
C13—Sn2—C19	136.73 (17)	Sn4—O2—Sn1	130.38 (14)
O2—Sn2—O1	74.26 (10)	Sn2—O2—Sn1	105.88 (12)

Disorder of the solvent molecule was modeled as two different orientations for Cl atoms. Refinement of the occupancy factors for atoms Cl5*A*, Cl6*A*, Cl5*B* and Cl6*B* revealed an approximate 85:15

ratio for the two components. Only the major component was refined anisotropically. All H atoms were initially located in a difference Fourier map. The phenyl and methylene H atoms were then constrained to an ideal geometry, with C—H distances in the range 0.93–0.97 Å and U_{iso}(H) = 0.08 Å^2.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SMART*; data reduction: *SAINT-Plus* (Bruker, 1999); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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