E. Mothi Mohamed,[a] Krishnaswamy Panchanatheswaran,[a] John N. Low[b]‡ and Christopher Glidewell[c]*

[a]Department of Chemistry, Bharathidasan University, Tiruchirappalli, Tamil Nadu 620 024, India, [b]Department of Chemistry, University of Aberdeen, Meston Walk, Old Aberdeen AB24 3UE, Scotland, and [c]School of Chemistry, University of St Andrews, Fife KY16 9ST, Scotland

‡ Postal address: Department of Electrical Engineering and Physics, University of Dundee, Dundee DD1 4HN, Scotland

Correspondence e-mail: cg@st-andrews.ac.uk

Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.007 Å
R factor = 0.038
wR factor = 0.088
Data-to-parameter ratio = 20.0

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

Octabenzyldichlorodi-μ2-hydroxo-di-μ3-oxo-tetratin toluene disolvate

The title tin complex crystallizes as a stoichiometric toluene-solvated dimer of 1,1,3,3-tetrabenzyl-1-chloro-3-hydroxydistannoxane, $[Sn_4(C_7H_7)_8Cl_2O_2(OH)_2]\cdot 2C_7H_8$. The tetranuclear molecule lies across a centre of inversion in space group $P2_1/n$; the Sn_4O_4 framework, in which the two independent Sn centres both have distorted trigonal bipyramidal coordination, is essentially planar.

Comment

The organotin component of the title compound, (I), was obtained as an adventitious product of the partial hydrolysis of dibenzyldichlorotin(IV) during the attempted preparation of adducts of this tin precursor complex with amines. It crystallizes from toluene as the stoichiometric disolvate 1,1,3,3-tetrabenzyl-1-chloro-3-hydroxydistannoxane–toluene (1/2).
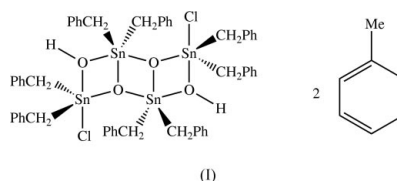

The tetranuclear molecule is a dimeric form of the simple distannoxane $ClSn(CH_2Ph)_2OSn(CH_2Ph)_2OH$. The dimer lies across a centre of inversion and the Sn_4O_4 framework, which is essentially planar, takes the form of three edge-fused Sn_2O_2 rings (Fig. 1 and Table 1). Each of the two independent Sn atoms is five-coordinate, adopting approximate trigonal
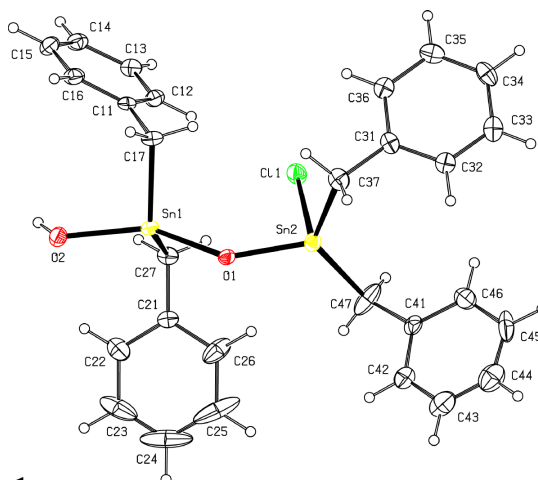
Figure 1
The asymmetric unit of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. For the sake of clarity, the toluene solvent molecule has been omitted.
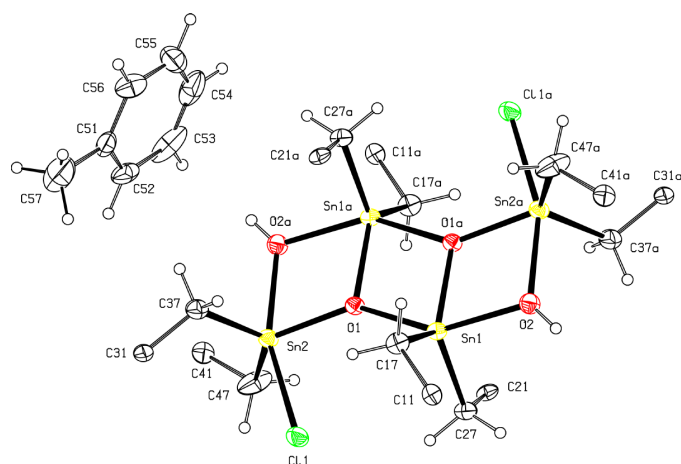
Figure 2
The centrosymmetric distannoxane dimer and the unique toluene solvent molecule. Displacement ellipsoids are drawn at the 30% probability level. For the sake of clarity, the phenyl ring atoms Cn2–Cn6 (n = 1–4) and the associated H atoms have been omitted. The atoms with the suffix a are at the symmetry position $(1 - x, 1 - y, 1 - z)$.

bipyramidal coordination, as demonstrated by the bond angles (Table 1): the benzyl groups occupy equatorial sites and the unique Cl bonded to Sn2 occupies an axial site.

This type of molecular architecture appears to be rather characteristic of functionalized distannoxanes XSnR_2OSnR_2Y, where X and Y are electron-rich substituents, such as OH, Cl, I or NCS, all of which are capable of coordinating to a second Sn atom (Chow, 1971; Puff *et al.*, 1981; Graziani *et al.*, 1983; Blair *et al.*, 1997; Dakternieks *et al.*, 1997; Lu *et al.*, 2001). The present example is unusual only inasmuch as the two electron-rich substituents X and Y are different, here Cl and OH; in most previously reported examples, these two substituents are the same, although an example with X = I and Y = OEt has been reported by Blair *et al.* (1997).

The Sn—O distances for the equatorial sites are slightly shorter than those for the axial sites (Table 1) and the Sn—C distances lie in the rather narrow range 2.144 (3)–2.156 (4) Å. There are no direction-specific interactions between the dimeric tin units. In particular, the hydroxyl group acts neither as a donor nor as an acceptor of hydrogen bonds: there are no O or C atoms within 3.6 Å of atom O2 other than those in the same dimer unit, and none of these is appropriately positioned to act as a hydrogen-bond donor or acceptor. It is likely that the hydroxyl group at (x, y, z) is effectively shielded by the adjacent benzyl substituents, especially by the phenyl rings C11–C16 and C21–C26 at (x, y, z) and C41–C46 at $(1 - x, 1 - y, 1 - z)$ (Figs. 1 and 2).

Experimental

The title distannoxane was obtained as an adventitious product from the attempted reaction of dibenzyldichlorotin(IV) with either 2-(dimethylamino)pyridine or N,N,N',N'-tetramethylethylenediamine. Crystallization from toluene solution gave the stoichiometric disolvate (I) in each case. Crystals of both were examined, and they proved to have identical cell dimensions; data sets were collected for both and they gave essentially identical refinements.

Table 1
Selected geometric parameters (Å, °).

Sn1—O1	2.132 (2)	Sn2—O1	2.024 (2)
Sn1—O2	2.147 (2)	Sn2—Cl1	2.4819 (10)
Sn1—C17	2.143 (3)	Sn2—C37	2.145 (4)
Sn1—C27	2.146 (3)	Sn2—C47	2.156 (4)
Sn1—O1^i	2.051 (2)	Sn2—O2^i	2.197 (3)
O1—Sn1—O2	146.07 (9)	Cl1—Sn2—O2^i	159.64 (7)
O1—Sn1—O1^i	73.34 (10)	Cl1—Sn2—O1	87.55 (7)
O1—Sn1—C17	100.30 (12)	Cl1—Sn2—C37	96.70 (12)
O1—Sn1—C27	99.02 (12)	Cl1—Sn2—C47	95.02 (13)
O2—Sn1—C17	97.73 (13)	O2^i—Sn2—O1	72.21 (10)
O2—Sn1—C27	96.07 (13)	O2^i—Sn2—C37	93.47 (13)
O2—Sn1—O1^i	72.76 (10)	O2^i—Sn2—C47	92.76 (15)
O1^i—Sn1—C17	120.11 (12)	O1—Sn2—C37	113.48 (13)
O1^i—Sn1—C27	119.09 (11)	O1—Sn2—C47	118.24 (18)
C17—Sn1—C27	120.70 (14)	C37—Sn2—C47	127.27 (19)
Sn1—O1—Sn2	140.75 (11)	Sn1^i—O1—Sn2	112.52 (11)
Sn1^i—O1—Sn1	106.66 (10)	Sn1—O2—Sn2^i	102.50 (11)
O2—Sn1—C17—C11	−62.5 (3)	O1—Sn2—C37—C31	−148.5 (3)
Sn1—C17—C11—C12	−69.0 (4)	Sn2—C37—C31—C32	−84.2 (4)
O2—Sn1—C27—C21	−81.4 (3)	O1—Sn2—C47—C41	−124.5 (3)
Sn1—C27—C21—C22	93.1 (4)	Sn2—C47—C41—C42	93.2 (4)

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

All H atoms were located from difference maps and then treated as riding atoms. H atoms bonded to C atoms were assigned C—H distances of 0.95 (aromatic) or 0.99 Å (CH$_2$), with U_{iso}(H) = 1.2U_{eq}(C); the H atom bonded to O2 was assigned an O—H distance of 0.84 Å and a U_{iso}(H) value of 1.2U_{eq}(O). The anisotropic displacement parameter values gave some indication of libration about Sn—C bonds in several of the benzyl groups: however, it did not prove possible to account for this using a static disorder model. The highest maximum is the diference map is adjacemt to the C45—C46 bond, 1.15 Å from C45 and 1.00 Å from C46; the deepest hole is 0.85 Å from Sn2.

Data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data

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