25.06. ^{119}Sn NMR in $CDCl_3$ = 103.1 p.p.m. (relative to tetramethyltin). The solid-state ^{119}Sn NMR spectrum was recorded on a Brüker MSL 200 spectrometer equipped with a double-bearing CP/MAS probe: ^{119}Sn NMR = −42.3 p.p.m. (relative to tetracyclohexyltin, δ = 176.0 p.p.m.). The solid-state ^{13}C NMR spectrum was also recorded, but some of the butyl and cyclohexyl signals could not be assigned because of extensive overlap. The spectrum showed three carbonyl (177.11, 177.49 and 179.28 p.p.m.) and three methylene (37.11, 35.32 and 33.58 p.p.m.) signals. The carbon signals were referenced to the carbonyl C atom of glycine (δ = 176.0 p.p.m.). Positive chemical shifts denote downfield shifts with respect to the standard.

Crystal data

$(C_{12}H_{24}N)_2[Sn(C_4H_4O_4)(C_4H_9)_3](C_4H_4O_4)_{1/2}.H_2O$
M_r = 846.79
Triclinic
$P\overline{1}$
a = 13.068 (3) Å
b = 13.715 (3) Å
c = 14.312 (3) Å
α = 84.48 (2)°
β = 65.25 (2)°
γ = 86.76 (2)°
V = 2318.4 (9) Å^3
Z = 2
D_x = 1.213 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 32 reflections
θ = 2.24–12.46°
μ = 0.596 mm^{-1}
T = 158 (2) K
Rhombic block
0.50 × 0.30 × 0.20 mm
Colorless

Data collection

Siemens P4 diffractometer
ω scan
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.843, T_{max} = 0.888
8129 measured reflections
8081 independent reflections
5229 reflections with $I > 2\sigma(I)$

R_{int} = 0.055
θ_{max} = 24.99°
$h = -15 \rightarrow 15$
$k = -16 \rightarrow 16$
$l = 0 \rightarrow 15$
3 standard reflections every 97 reflections intensity decay: 6.84%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.069
$wR(F^2)$ = 0.160
S = 1.032
8080 reflections
477 parameters
H atoms riding, U(H) = 1.5U_{eq}(C,N); water H atoms located and refined

$w = 1/[\sigma^2(F_o^2) + (0.0506P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.665 e Å^{-3}
$\Delta\rho_{min}$ = −0.555 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Sn1—C1	2.123 (7)	N1···O6	2.749 (7)
Sn1—C5	2.156 (7)	N2···O5	2.709 (7)
Sn1—C9	2.132 (7)	N2···O7^i	2.771 (9)
Sn1—O1	2.230 (5)	O2···O7	2.733 (8)
Sn1—O3	2.279 (5)	O6···O7	2.775 (8)
N1···O4	2.743 (7)		
C1—Sn1—C5	112.4 (3)	C5—Sn1—O1	88.0 (2)
C1—Sn1—C9	134.4 (3)	C5—Sn1—O3	87.6 (2)
C1—Sn1—O1	93.3 (2)	C9—Sn1—O1	90.2 (2)
C1—Sn1—O3	91.4 (2)	C9—Sn1—O3	88.6 (2)
C5—Sn1—C9	113.1 (3)	O1—Sn1—O3	174.5 (2)

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

Data collection: *XSCANS* (Siemens, 1991). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SX1058). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1998). C**54**, 752–755

Octabutyl-1κ^2C,2κ^2C,3κ^2C,4κ^2C-bis-μ_3-oxo-1:2:3κ^3O;1:3:4κ^3O-tetrakis[μ-(2-pyridinecarboxylato N-oxide)]-1κ^2O,O':2κO;2κO:3κ^2O,O';3κ^2O,O':4κO;1κ^2O,O':4κO-tetratin Hydrate

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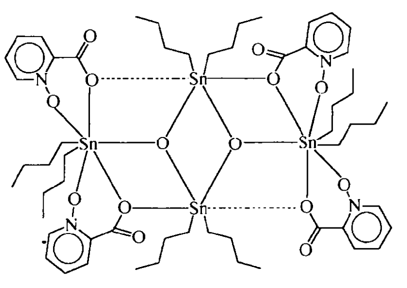
Abstract

The unit cell of the title compound, bis[1,1,3,3-tetrabutyl-1,3-bis(picolinato N-oxide)distannoxane] hy-

drate, $[Sn_4(O)_2(C_4H_9)_8(C_6H_4NO_3)_4].H_2O$, contains two independent centrosymmetric dicarboxylatotetraorganodistannoxane dimers, one of which is hydrogen bonded to two water molecules. Both dimers possess a planar Sn_2O_2 core unit, the O atoms of which are covalently bonded to two [dibutyl(picolinato *N*-oxide)tin] units. The endocyclic Sn atoms (of the Sn_2O_2 core units) are six-coordinate in C_2SnO_4 skew-trapezoidal bipyramidal environments whereas the exocyclic Sn atoms {of the [dibutyl(picolinato *N*-oxide)tin] units} are seven-coordinate in *trans*-C_2SnO_5 pentagonal bipyramidal environments.

Comment

Dimeric dicarboxylatotetrabutyldistannoxanes adopt centrosymmetric structures that are characterized by Sn1–O1–Sn2–O5 and Sn1′–O1′–Sn2′–O5′ rings flanking an Sn2–O1–Sn2′–O1′ core on opposite sides (Ng *et al.*, 1991, 1994). (The primed atoms are related to the unprimed atoms by a center of symmetry.) In the picolinato (2-pyridinecarboxylato) derivative, the exocyclic Sn atom (*i.e.* Sn1) is six-coordinate, arising from bidentate chelation by one picolinato anion [Sn—O = 2.134 (4) and Sn—N = 2.550 (5) Å] and bonding by an additional monodentate picolinato anion [Sn—O = 2.303 (4) Å]. However, the geometry is distorted towards pentagonal bipyramidal owing to a weak Sn···N interaction [3.150 (5) Å]. The endocyclic Sn atom (*i.e.* Sn2) is essentially five-coordinate in a *cis*-trigonal bipyramidal environment, but the coordination sphere is distorted towards a skew-trapezoidal bipyramid (Parulekar *et al.*, 1989). The inability of the exocyclic Sn atom to achieve seven-coordination appears to arise largely from steric effects as a shorter interaction would draw the two pyridyl rings too close together. This possibility is avoided in the picolinato *N*-oxide derivative, (I), as the oxide O atom can coordinate to the Sn atom. The compound crystallizes as two symmetry-independent molecules, one of which is hydrogen bonded [O···O = 2.97 (2) Å] to two molecules of water.



(I)

The exocyclic Sn atom is chelated by two picolinato groups through the *N*-oxide and carboxyl ends in a seven-coordinate geometry. The Sn—$O_{N\text{-oxide}}$ bonds comprise a short and a long bond [molecule *a*: 2.336 (5) and 2.506 (4) Å; molecule *b*: 2.311 (5), 2.593 (5); 2.369 (5) and 2.507 (5) Å (molecule *b*, although centrosymmetric, is disordered, which gives rise to two sets of bonds)]. The shorter distance of each pair is similar to intermolecular Sn—O distances found in the isomeric triphenyltin 2-, 3- and 4-pyridinecarboxylate *N*-oxides (Ng & Kumar Das, 1995). In both molecules, the $-CO_2$ plane is twisted relative to the $-C_5H_4N$ plane to enable the picolinato groups to chelate [molecule *a*: dihedral angle = 21.4 (3) and 42.0 (5)°; molecule *b*: dihedral angle = 40.4 (7) and 70 (1)°].

Hydrogen-bonding interactions involving water in organotin hydrates are usually extensive (Kumar Das *et al.*, 1987), but only one such interaction [O7*a*···O8*a* = 2.97 (2) Å] is present in the distannoxane, which explains why the crystals turn opaque when exposed to air as the lattice water escapes from the structure. The water is disordered, but only the O8*a* half participates in hydrogen bonding. The anhydrous compound probably adopts an essentially identical solid-state structure.

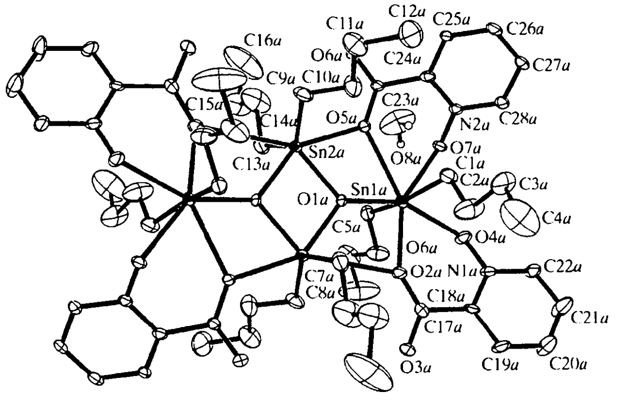


Fig. 1. Plot of molecule *a* of bis[tetrabutylbis(picolinato *N*-oxide)distannoxane] hydrate at the 25% probability level. H atoms are not shown except for those of water.

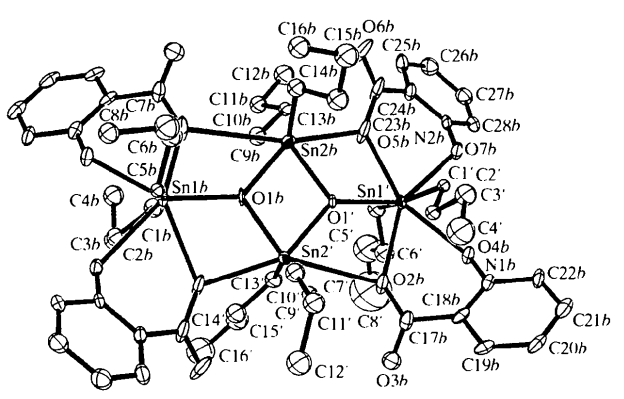


Fig. 2. Plot of molecule *b* of bis[tetrabutylbis(picolinato *N*-oxide)distannoxane] hydrate at the 25% probability level. H atoms are not shown.

The ^{119m}Sn Mössbauer spectrum of the anhydrous compound consists of two overlapping doublets. The doublet having the larger quadrupole splitting (4.06 mm s^{-1}) is assigned to the exocyclic Sn atom [molecule *a*: C—Sn—C = 165.0 (3)°; molecule *b*: C—Sn—C = 163.4 (6) and 168.3 (6)°] whereas the doublet having the smaller value (2.92 mm s^{-1}) is assigned to the endocyclic Sn atom [molecule *a*: C—Sn—C = 131.8 (3)°; molecule *b*: C—Sn—C = 131.0 (7) and 133.3 (6)°], in accordance with point-charge approximations (Sham & Bancroft, 1975) that relate the observed quadrupole splittings to the C—Sn—C angles.

Experimental

Equimolar amounts of dibutyltin oxide and picolinic acid *N*-oxide were heated in a small volume of ethanol until the oxide dissolved completely. The filtered solution yielded large crystals when the solvent was allowed to evaporate slowly, but the crystals rapidly turned opaque when taken out of solution. The opaque material was purified by dissolution in chloroform followed by precipitation with hexane to give a white powder. The white powder was formulated from its elemental analysis as anhydrous bis[1,1,3,3-tetrabutyl-1,3-bis(picolinato *N*-oxide)-distannoxane]. Found: C 44.60, H 5.85, N 3.78%; calculated for $C_{56}H_{88}N_4O_{14}$: C 44.37, H 5.85, N 3.70%. The compound did not have a clear melting point; it began to char at about 458 K. IR (Nujol): 1656 (asym. CO_2), 1209, 1160 cm^{-1} (N→O) [*cf.* picolinic acid *N*-oxide: 1679 (asym. CO_2), 1286, 1253 cm^{-1} (N→O)]. ^{119m}Sn Mössbauer (78 K): doublet, isomer shift 1.32, quadrupole splitting 4.06, τ 0.96 mm^{-1}; doublet, isomer shift 1.24, quadrupole splitting 2.92 mm s^{-1}, τ 0.98 mm^{-1} (ratio of doublet areas = 49/51) [the respective literature values are 1.29, 3.92; 1.17, 2.95 mm s^{-1} (Sandhu & Boparoy, 1991)]. Crystals of the hydrated compound for the diffraction measurements were obtained by the slow evaporation of an ethanol solution containing dissolved dibutyltin oxide and picolinic acid *N*-oxide in 1:1 molar amounts.

Crystal data

$[Sn_4(O)_2(C_4H_9)_8(C_6H_4NO_3)_4].H_2O$
$M_r = 1534.08$
Triclinic
$P\bar{1}$
$a = 12.442\ (2)$ Å
$b = 12.516\ (3)$ Å
$c = 22.163\ (5)$ Å
$\alpha = 88.01\ (6)°$
$\beta = 78.48\ (3)°$
$\gamma = 77.88\ (1)°$
$V = 3306\ (1)$ Å^3
$Z = 2$
$D_x = 1.541$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 2.0–12.5°$
$\mu = 1.555$ mm^{-1}
$T = 158\ (2)$ K
Triangular block
0.50 × 0.30 × 0.30 mm
Colorless

Data collection

Siemens *P*4 diffractometer
ω scan
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.577$, $T_{\max} = 0.627$
11 702 measured reflections
11 647 independent reflections

7546 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.030$
$\theta_{\max} = 25.01°$
$h = -14 \rightarrow 0$
$k = -14 \rightarrow 14$
$l = -26 \rightarrow 25$
3 standard reflections every 97 reflections intensity decay: 12.25%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.133$
$S = 0.900$
11647 reflections
684 parameters
H atoms riding, U(H) = $1.5U_{\rm eq}$(C); H atoms of disordered water were placed at calculated positions

$w = 1/[\sigma^2(F_o^2) + (0.0782P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 1.109$ e Å^{-3}
$\Delta\rho_{\min} = -0.966$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Sn1*a*—C1*a*	2.128 (8)	Sn2*b*—C9*b*	2.19 (2)
Sn1*a*—C5*a*	2.119 (8)	Sn2*b*—C13*b*	2.08 (2)
Sn1*a*—O1*a*	2.102 (4)	Sn2*b*—O1*b*	2.10 (1)
Sn1*a*—O2*a*	2.184 (5)	Sn2*b*—O1′	2.04 (1)
Sn1*a*—O4*a*	2.336 (5)	Sn2*b*—O2$b^{\rm ii}$	2.895 (5)
Sn1*a*—O5*a*	2.468 (5)	Sn2*b*—O5*b*	2.407 (5)
Sn1*a*—O7*a*	2.506 (4)	Sn1′—C1′	2.03 (1)
Sn2*a*—C9*a*	2.085 (7)	Sn1′—C5′	2.12 (2)
Sn2*a*—C13*a*	2.142 (9)	Sn1′—O1′	2.089 (9)
Sn2*a*—O1*a*	2.025 (4)	Sn1′—O2*b*	2.390 (7)
Sn2*a*—O1$a^{\rm i}$	2.093 (4)	Sn1′—O4*b*	2.507 (5)
Sn2*a*—O2$a^{\rm i}$	3.083 (5)	Sn1′—O5*b*	2.273 (6)
Sn2*a*—O5*a*	2.360 (4)	Sn1′—O7*b*	2.369 (5)
Sn1*b*—C1*b*	2.08 (2)	Sn2′—C9′	2.10 (2)
Sn1*b*—C5*b*	2.11 (1)	Sn2′—C13′	2.12 (2)
Sn1*b*—O1*b*	2.086 (9)	Sn2′—O1′	2.088 (9)
Sn1*b*—O2$b^{\rm ii}$	2.097 (6)	Sn2′—O1*b*	2.05 (1)
Sn1*b*—O4$b^{\rm ii}$	2.311 (5)	Sn2′—O2*b*	2.950 (6)
Sn1*b*—O5$b^{\rm ii}$	2.571 (7)	Sn2′—O5$b^{\rm ii}$	2.454 (6)
Sn1*b*—O7$b^{\rm ii}$	2.593 (5)		
C1*a*—Sn1*a*—C5*a*	165.0 (3)	O5$b^{\rm ii}$—Sn1*b*—O7$b^{\rm ii}$	64.7 (2)
C1*a*—Sn1*a*—O1*a*	96.3 (3)	C9*b*—Sn2*b*—C13*b*	131.0 (7)
C1*a*—Sn1*a*—O2*a*	95.8 (2)	C9*b*—Sn2*b*—O5*b*	85.4 (5)
C1*a*—Sn1*a*—O4*a*	88.7 (3)	C9*b*—Sn2*b*—O1*b*	98.5 (5)
C1*a*—Sn1*a*—O5*a*	87.5 (3)	C9*b*—Sn2*b*—O1′	109.4 (7)
C1*a*—Sn1*a*—O7*a*	84.5 (2)	C9*b*—Sn2*b*—O2$b^{\rm ii}$	72.4 (5)
C5*a*—Sn1*a*—O1*a*	94.0 (2)	C13*b*—Sn2*b*—O1*b*	106.8 (6)
C5*a*—Sn1*a*—O2*a*	97.0 (3)	C13*b*—Sn2*b*—O1′	117.0 (5)
C5*a*—Sn1*a*—O4*a*	87.0 (2)	C13*b*—Sn2*b*—O2$b^{\rm ii}$	87.1 (5)
C5*a*—Sn1*a*—O5*a*	86.6 (2)	C13*b*—Sn2*b*—O5*b*	98.7 (5)
C5*a*—Sn1*a*—O7*a*	80.6 (2)	O1*b*—Sn2*b*—O2$b^{\rm ii}$	57.1 (3)
O1*a*—Sn1*a*—O2*a*	77.6 (2)	O1*b*—Sn2*b*—O5*b*	141.3 (4)
O1*a*—Sn1*a*—O4*a*	153.1 (2)	O1*b*—Sn2*b*—O1′	76.4 (4)
O1*a*—Sn1*a*—O5*a*	67.3 (2)	O1′—Sn2*b*—O2$b^{\rm ii}$	132.4 (3)
O1*a*—Sn1*a*—O7*a*	134.1 (2)	O1′—Sn2*b*—O5*b*	66.5 (3)
O2*a*—Sn1*a*—O4*a*	75.6 (2)	O2$b^{\rm ii}$—Sn2*b*—O5*b*	154.5 (2)
O2*a*—Sn1*a*—O5*a*	144.8 (2)	Sn1*b*—O1*b*—Sn2*b*	131.4 (5)
O2*a*—Sn1*a*—O7*a*	148.2 (2)	Sn1*b*—O1*b*—Sn2′	123.4 (5)
O4*a*—Sn1*a*—O5*a*	139.5 (2)	Sn2*b*—O1*b*—Sn2′	103.1 (4)
O4*a*—Sn1*a*—O7*a*	72.6 (2)	C1′—Sn1′—C5′	163.4 (6)
O5*a*—Sn1*a*—O7*a*	66.9 (2)	C1′—Sn1′—O1′	98.1 (5)
C9*a*—Sn2*a*—C13*a*	131.8 (3)	C1′—Sn1′—O2*b*	103.2 (4)
C9*a*—Sn2*a*—O1*a*	114.3 (3)	C1′—Sn1′—O4*b*	87.0 (4)
C9*a*—Sn2*a*—O1$a^{\rm i}$	103.7 (3)	C1′—Sn1′—O5*b*	98.6 (4)
C9*a*—Sn2*a*—O2$a^{\rm i}$	72.3 (2)	C1′—Sn1′—O7*b*	79.8 (4)
C9*a*—Sn2*a*—O5*a*	92.8 (2)	C5′—Sn1′—O1′	96.5 (6)
C13*a*—Sn2*a*—O1*a*	112.2 (3)	C5′—Sn1′—O2*b*	88.1 (5)
C13*a*—Sn2*a*—O1$a^{\rm i}$	98.6 (3)	C5′—Sn1′—O4*b*	86.0 (5)

C13*a*—Sn2*a*—O2*a*^{i}	84.2 (3)	C5′—Sn1′—O5*b*	79.6 (5)
C13*a*—Sn2*a*—O5*a*	90.8 (3)	C5′—Sn1′—O7*b*	84.0 (5)
O1*a*—Sn2*a*—O1*a*^{i}	76.6 (2)	O1′—Sn1′—O2*b*	75.2 (3)
O1*a*—Sn2*a*—O2*a*^{i}	134.7 (2)	O1′—Sn1′—O4*b*	142.8 (3)
O1*a*—Sn2*a*—O5*a*	70.6 (2)	O1′—Sn1′—O5*b*	68.5 (3)
O1*a*^{i}—Sn2*a*—O2*a*^{i}	58.9 (2)	O1′—Sn1′—O7*b*	140.7 (3)
O1*a*^{i}—Sn2*a*—O5*a*	147.1 (2)	O2*b*—Sn1′—O4*b*	67.8 (2)
O2*a*^{i}—Sn2*a*—O5*a*	154.0 (1)	O2*b*—Sn1′—O5*b*	139.8 (2)
Sn1*a*—O1*a*—Sn2*a*	123.2 (2)	O2*b*—Sn1′—O7*b*	143.8 (2)
Sn1*a*—O1*a*—Sn2*a*^{i}	131.4 (2)	O4*b*—Sn1′—O5*b*	147.3 (2)
Sn2*a*—O1*a*—Sn2*a*^{i}	103.4 (2)	O4*b*—Sn1′—O7*b*	76.5 (2)
C1*b*—Sn1*b*—C5*b*	168.3 (6)	O5*b*—Sn1′—O7*b*	73.0 (2)
C1*b*—Sn1*b*—O1*b*	92.7 (6)	C9′—Sn2′—C13′	133.3 (6)
C1*b*—Sn1*b*—O2*b*^{ii}	79.6 (6)	C9′—Sn2′—O1′	107.4 (6)
C1*b*—Sn1*b*—O4*b*^{ii}	86.1 (6)	C9′—Sn2′—O1*b*	115.9 (5)
C1*b*—Sn1*b*—O5*b*^{ii}	85.2 (6)	C9′—Sn2′—O2*b*	82.1 (4)
C1*b*—Sn1*b*—O7*b*^{ii}	85.6 (5)	C9′—Sn2′—O5*b*^{ii}	89.9 (5)
C5*b*—Sn1*b*—O1*b*	98.9 (5)	C13′—Sn2′—O1′	98.4 (5)
C5*b*—Sn1*b*—O2*b*^{ii}	105.1 (4)	C13′—Sn2′—O1*b*	107.6 (6)
C5*b*—Sn1*b*—O4*b*^{ii}	84.6 (4)	C13′—Sn2′—O2*b*	75.6 (5)
C5*b*—Sn1*b*—O5*b*^{ii}	97.4 (4)	C13′—Sn2′—O5*b*^{ii}	86.7 (4)
C5*b*—Sn1*b*—O7*b*^{ii}	85.2 (4)	O1′—Sn2′—O1*b*	76.2 (4)
O1*b*—Sn1*b*—O2*b*^{ii}	73.0 (3)	O1′—Sn2′—O2*b*	63.3 (3)
O1*b*—Sn1*b*—O4*b*^{ii}	149.1 (3)	O1′—Sn2′—O5*b*^{ii}	149.3 (3)
O1*b*—Sn1*b*—O5*b*^{ii}	70.3 (3)	O1*b*—Sn2′—O2*b*	139.3 (3)
O1*b*—Sn1*b*—O7*b*^{ii}	134.9 (3)	O1*b*—Sn2′—O5*b*^{ii}	73.4 (3)
O2*b*^{ii}—Sn1*b*—O4*b*^{ii}	76.4 (2)	O2*b*—Sn2′—O5*b*^{ii}	146.2 (2)
O2*b*^{ii}—Sn1*b*—O5*b*^{ii}	139.4 (2)	Sn1′—O1′—Sn2′	131.1 (5)
O2*b*^{ii}—Sn1*b*—O7*b*^{ii}	149.2 (2)	Sn1′—O1′—Sn2*b*	123.1 (5)
O4*b*^{ii}—Sn1*b*—O5*b*^{ii}	140.0 (2)	Sn2′—O1′—Sn2*b*	103.6 (4)
O4*b*^{ii}—Sn1*b*—O7*b*^{ii}	75.8 (2)		

Symmetry codes: (i) $-x, 1-y, -z$; (ii) $1-x, -y, 1-z$.

The $(C_4H_9)_2Sn$ portion of molecule *b* is ordered over a center of inversion and was refined as two $[(C_4H_9)_2SnO]$ units, each of half site occupancy; their C atoms were refined isotropically. The pyridyl rings were refined as rigid hexagons. The C atoms of the butyl groups of the *a* and *b* molecules were refined subject to 1,2- and 1,3-related atom distances of 1.54±0.01 and 2.52±0.02 Å, respectively; restraints were imposed on the atoms of the chains. The final difference map had peaks of about 1 e Å^{-3} near the Sn atoms.

Data collection: *XSCANS* (Siemens, 1990). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SX1060). Services for accessing these data are described at the back of the journal.

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Racemic 1,1,6,6-Tetramethyl-1,6-distannatetranaphtho[2,1-*b*;1′,2′-*d*;2″,1″-*g*;1‴,2‴-*i*]cyclodeca-2,4,7,9-tetraene

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Abstract

The title compound, $[Sn_2(C_{20}H_{12})_2(CH_3)_4]$, which is the first chiral tin-bridged cyclophane, was prepared from the reaction of racemic 2,2′-dilithio-1,1′-binaphthyl and dimethyltin dichloride. The molecule has approximate local D_2 222 symmetry.

Comment

An optically active compound containing the 1,1′-binaphthyl moiety is one of the potential target molecules for non-linear optical materials (Thorup, 1996). Much attention is being devoted to compounds containing a 2,2′-disubstituted 1,1′-biaryl group as efficient ligands of asymmetric catalysts or chiral reagents in asymmetric syntheses (Kagan, 1985; Takaya *et al.*, 1993; Noyori, 1994). As new examples of such chiral compounds, the authors and others have recently independently reported the preparation, structural analyses and properties of dinaphtho[2,1-*b*;1′,2′-*d*]phosphole derivatives (Tani, Yamagata, & Tashiro, 1995; Tani, Tashiro *et al.*, 1995; Gladiali *et al.*, 1994) and their arsenic