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Octabutyl-1κ^2C,2κ^2C,3κ^2C,4κ^2C-di-μ_3-oxo-1:2:3κ^3O;2:3:4κ^2O-di-μ_2-phenoxy-1:2κ^2O;3:4κ^2O-diphenoxy-1κO,4κO-tetratin(IV)

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Key indicators

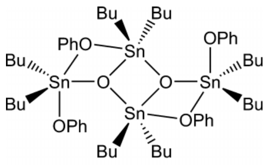
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.007 Å
Disorder in main residue
R factor = 0.025
wR factor = 0.044
Data-to-parameter ratio = 20.0

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

The dimeric title compound, tetrabutyldiphenoxydistannoxane, [$Sn_4(C_4H_9)_8(C_6H_5O)_4O_2$], adopts a ladder-type structure, featuring an almost planar inorganic framework with three four-membered Sn_2O_2 rings and four coplanar phenoxy groups.

Comment

The title compound, [$Bu_2(PhO)SnOSn(OPh)Bu_2]_2$, (I), was obtained by the condensation of dibutyltin oxide with phenol according to a literature procedure (Considine *et al.*, 1963). The structure of (I) (Fig. 1 and Table 1) features an almost planar $Sn_4O_2(OPh)_4$ motif, the largest deviation from the mean plane being 0.072 (1) Å for Sn4. The four independent Sn atoms adopt a distorted trigonal bipyramidal geometry defined by SnC_2O_3 donor sets. The distortion is more pronounced at the exocyclic Sn atoms Sn1 and Sn4 than at the endocyclic atoms Sn2 and Sn3 [geometrical goodness $\Delta\Sigma(\theta)$: 77.8° for Sn1, 85.8° for Sn2, 85.3° for Sn3, and 78.7° for Sn4] (Kolb *et al.*, 1994). The ^{119}Sn MAS NMR spectrum of (I) shows two signals at δ_{iso} −175.8 (integral 56%) and −191.6 (integral 44%), which are assigned to the endocyclic and exocyclic Sn atoms, respectively. Despite being crystallographically inequivalent, the differences between Sn1 and Sn4 or Sn2 and Sn3 are apparently too marginal to be resolved by ^{119}Sn MAS NMR spectroscopy. Overall, the structure of (I) closely resembles those of many known dimeric tetraorganodistannoxanes, as reviewed by Davies (2004).



(I)

Experimental

Compound (I) was prepared by the method of Considine *et al.* (1963) and recrystallized from dichloromethane/hexane (1:1, 1 g/20 ml) at room temperature.

Crystal data

[$Sn_4(C_4H_9)_8(C_6H_5O)_4O_2$]
M_r = 1336.06
Monoclinic, $P2_1$
a = 13.1456 (4) Å
b = 17.1448 (6) Å
c = 13.3802 (6) Å
β = 101.070 (2)°
V = 2959.50 (19) Å^3
Z = 2
D_x = 1.499 Mg m^{-3}

Mo *Kα* radiation
Cell parameters from 24855 reflections
θ = 3.1–27.5°
μ = 1.71 mm^{-1}
T = 173 (2) K
Block, colourless
0.35 × 0.25 × 0.20 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{\min} = 0.562$, $T_{\max} = 0.715$
24855 measured reflections
12278 independent reflections
9730 reflections with $I > 2\sigma(I)$
$\theta_{\max} = 27.5^\circ$
$h = -16 \rightarrow 16$
$k = -21 \rightarrow 22$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.026$
$wR(F^2) = 0.044$
$S = 0.87$
12278 reflections
613 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2)]$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.59$ e Å^{-3}
$\Delta\rho_{\min} = -0.45$ e Å^{-3}
Absolute structure: Flack (1983), 5263 Friedel pairs
Flack parameter = 0.394 (14)

Table 1
Selected geometric parameters (Å, °).

Sn1—C1	2.119 (4)	Sn3—C31	2.133 (4)
Sn1—C11	2.127 (4)	Sn3—C35	2.138 (4)
Sn1—O1	1.999 (2)	Sn3—O1	2.043 (2)
Sn1—O3	2.315 (2)	Sn3—O2	2.107 (2)
Sn1—O5	2.096 (2)	Sn3—O3	2.274 (2)
Sn2—C21	2.102 (5)	Sn4—C41	2.140 (4)
Sn2—C25	2.134 (4)	Sn4—C51	2.130 (4)
Sn2—O1	2.098 (2)	Sn4—O2	1.998 (2)
Sn2—O2	2.043 (2)	Sn4—O4	2.315 (2)
Sn2—O4	2.275 (2)	Sn4—O6	2.092 (2)
Sn1—O1—Sn3	120.25 (12)	O2—Sn2—O4	70.26 (9)
Sn1—O1—Sn2	131.61 (11)	O1—Sn2—O4	142.25 (9)
Sn3—O1—Sn2	108.08 (11)	C21—Sn2—O4	91.48 (15)
Sn4—O2—Sn2	120.09 (12)	C25—Sn2—O4	95.70 (14)
Sn4—O2—Sn3	131.74 (11)	O1—Sn3—O2	71.99 (9)
Sn2—O2—Sn3	107.75 (11)	O1—Sn3—C31	111.92 (12)
Sn3—O3—Sn1	99.60 (9)	O2—Sn3—C31	99.28 (13)
Sn2—O4—Sn4	99.44 (9)	O1—Sn3—C35	117.07 (13)
O1—Sn1—O5	78.00 (10)	O2—Sn3—C35	102.75 (14)
O1—Sn1—C1	113.85 (14)	C31—Sn3—C35	130.35 (15)
O5—Sn1—C1	103.18 (13)	O1—Sn3—O3	70.13 (9)
O1—Sn1—C11	116.19 (13)	O2—Sn3—O3	142.08 (8)
O5—Sn1—C11	99.28 (13)	C31—Sn3—O3	96.25 (13)
C1—Sn1—C11	128.22 (17)	C35—Sn3—O3	92.83 (14)
O1—Sn1—O3	70.01 (9)	O2—Sn4—O6	78.58 (9)
O5—Sn1—O3	148.00 (9)	O2—Sn4—C51	115.92 (14)
C1—Sn1—O3	89.25 (12)	O6—Sn4—C51	101.91 (13)
C11—Sn1—O3	95.57 (12)	O2—Sn4—C41	113.83 (13)
O2—Sn2—O1	72.17 (9)	O6—Sn4—C41	99.26 (13)
O2—Sn2—C21	114.97 (15)	C51—Sn4—C41	128.69 (17)
O1—Sn2—C21	101.10 (17)	O2—Sn4—O4	70.14 (9)
O2—Sn2—C25	109.63 (13)	O6—Sn4—O4	148.59 (9)
O1—Sn2—C25	100.05 (15)	C51—Sn4—O4	89.52 (12)
C21—Sn2—C25	134.59 (18)	C41—Sn4—O4	95.90 (12)

H atoms were placed in geometrically calculated positions and refined using a riding model (C—H$_{\text{prim.}}$ 0.96 Å, C—H$_{sec.}$ 0.97 Å, C—H$_{tert.}$ 0.93 Å). The isotropic displacement parameters were constrained to 1.2 (for non methyl) and 1.5 (for methyl groups) times U_{eq} of the carrier C atom. The crystal studied is racemically twinned, in a 0.394 (14):0.606 (14) ratio. Disorder was resolved for one butyl group so that C22 and C23 were each refined over two sites with occupancy factors of 0.6:0.4 and were fixed at this ratio in the last refinement cycle.

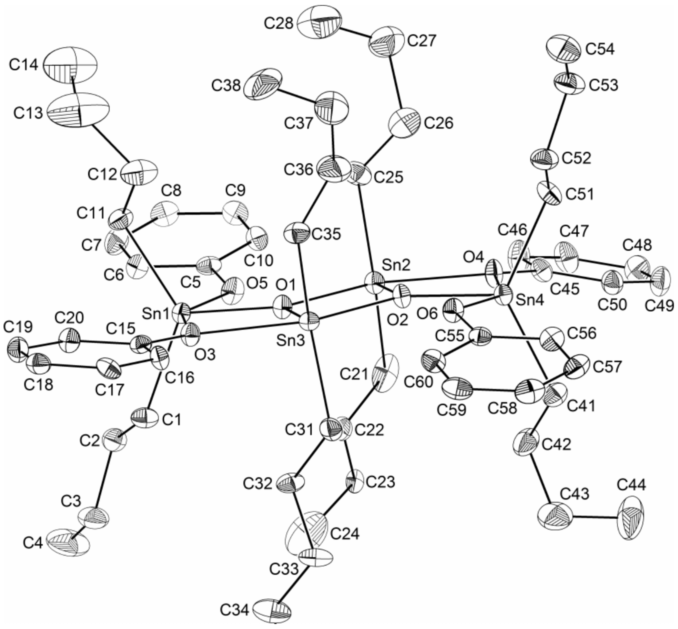


Figure 1
View of (I), showing the labelling of all non-H atoms. Displacement ellipsoids are shown at the 30% probability level. Minor disorder components of the C atoms and all H atoms have been omitted for clarity.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2000); software used to prepare material for publication: *SHELXL*97.

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