

Hexameric trimethylsilylmethyloxotin acetate, $[(\text{Me}_3\text{SiCH}_2)\text{Sn}(\text{O})(\text{OAc})]_6$

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Key indicators

Single-crystal X-ray study

$T = 223 \text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.006 \text{ \AA}$

R factor = 0.044

wR factor = 0.093

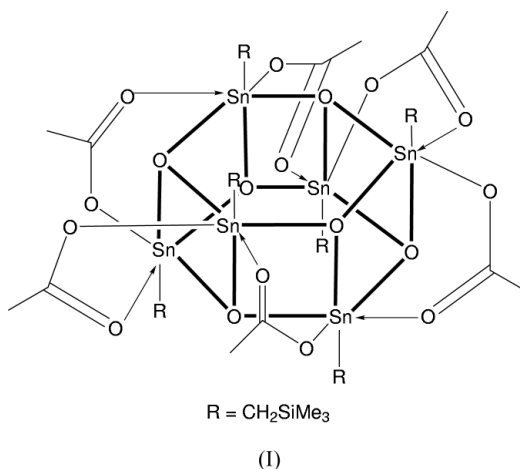
Data-to-parameter ratio = 31.5

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

The centrosymmetric hexanuclear title compound, hexa- μ_2 -acetato-hexa- μ_3 -oxo-hexakis[(trimethylsilylmethyl)tin], $[\text{Sn}_6\text{O}_6(\text{C}_2\text{H}_3\text{O}_2)_6(\text{C}_4\text{H}_{11}\text{Si})_6]$, adopts a 'drum' structure in which two $[(\text{Me}_3\text{SiCH}_2)\text{SnO}]_3$ caps are linked to each other via six μ_3 -O atoms and six bidentate bridging acetate groups. A CO_5 donor set defines a distorted octahedral environment for each of the three independent Sn atoms.

Comment

The title compound, (I), was obtained as a hydrolysis product of $(\text{Me}_3\text{SiCH}_2)_2\text{Sn}(\text{OAc})_2$ (see *Experimental*). The structure (Fig. 1 and Table 1) adopts a hexameric drum motif as found previously for related species (see reviews: Tiekink, 1991, 1994).



The centrosymmetric structure may be described as comprising two $[(\text{Me}_3\text{SiCH}_2)\text{SnO}]_3$ rings that are linked in two complementary ways. Each of the three bridging oxo atoms of each ring connects to an Sn atom of the second ring, of opposite orientation, leading to the presence of six μ_3 -oxo groups. The girth of the drum may be considered thus as six Sn_2O_2 rectangles arranged so as to form a tube. Six bidentate bridging acetate bridges afford additional links between the faces but, in this case, these diagonally span a Sn_2O_2 rectangle. The Me_3SiCH_2 groups are oriented above and below the faces of the drum. A distorted octahedral CO_5 geometry, defined by three O atoms derived from three μ_3 -O atoms, two carboxylate O atoms and a C atom of the Sn-bound substituent, is found for each Sn atom.

Experimental

The title compound, (I), was obtained as single crystals from a solution of authenticated $(\text{Me}_3\text{SiCH}_2)_2\text{Sn}(\text{OAc})_2$, presumably as a

Received 4 May 2004

Accepted 6 May 2004

Online 15 May 2004

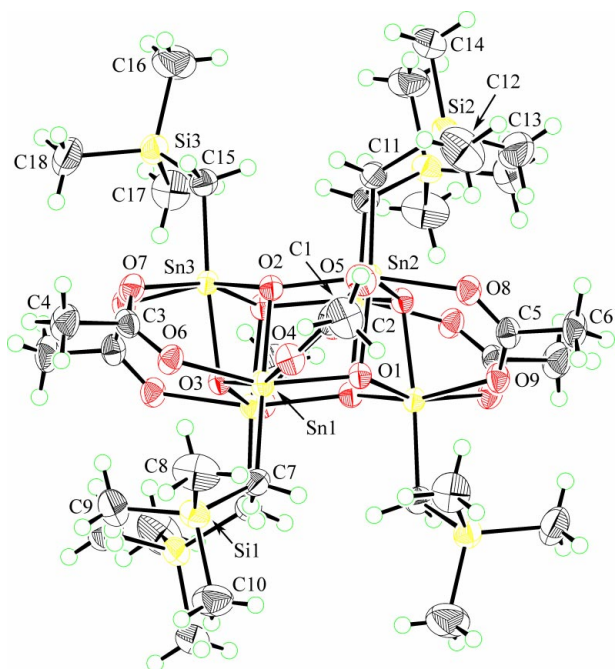


Figure 1
The molecular structure and crystallographic numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level (Johnson, 1976).

result of cleavage of the Me_3SiCH_2 group followed by hydrolysis upon standing. A solution of $(\text{Me}_3\text{SiCH}_2)_2\text{SnPh}_2$ (10.00 g, 23.9 mmol) in Et_2O (100 ml) and concentrated acetic acid (80 ml) was stirred overnight under reflux. The organic layer was collected and the aqueous layer washed with additional Et_2O (50 ml). The combined organic layer was dried over Na_2SO_4 , filtered and the solvent removed *in vacuo* to give $(\text{Me}_3\text{SiCH}_2)_2\text{Sn}(\text{OAc})_2$ as a clear liquid (7.90 g, 86% yield). ^1H NMR (299.98 MHz, CDCl_3): δ -0.06 (s, 18H, SiMe_3), 0.47 [s, $^2J(^1\text{H}-^{117/119}\text{Sn}) = 100/105$, 4H, CH_2], 1.90 (s, 6H, O_2CMe); $^{13}\text{C}\{^1\text{H}\}$ NMR (75.44 MHz, CDCl_3): δ 0.55 [$^3J(^{13}\text{C}-^{117/119}\text{Sn}) = 28$, SiMe_3], 9.22 [$^1J(^{13}\text{C}-^{117/119}\text{Sn}) = 463/485$, CH_2], 20.63 (O_2CMe), 180.38 ($\text{C}=\text{O}$); $^{119}\text{Sn}\{^1\text{H}\}$ NMR (111.85 MHz, CDCl_3): δ -118.5. Analysis calculated for $\text{C}_{12}\text{H}_{28}\text{O}_4\text{Si}_2\text{Sn}$ ($M_r = 411.23$): C 35.05, H 6.86%; found: C 34.70, H 7.03%. Hexane (15 ml) was added to the $(\text{Me}_3\text{SiCH}_2)_2\text{Sn}(\text{OAc})_2$ and the solution allowed to stand at room temperature for 2 d. Colourless crystals of $[(\text{Me}_3\text{SiCH}_2)_2\text{Sn}(\text{O})(\text{OAc})_6]$ were collected (0.37 g, 6% yield, m.p. 565–567 K). ^1H NMR (399.78 MHz, CDCl_3): δ 0.08 (s, 9H, SiMe_3), 0.17 [s, $^2J(^1\text{H}-^{117/119}\text{Sn}) = 153/159$, 2H, CH_2], 1.97 (s, 3H, O_2CMe); $^{13}\text{C}\{^1\text{H}\}$ NMR (75.44 MHz, CDCl_3): δ 1.20 [$^3J(^{13}\text{C}-^{117/119}\text{Sn}) = 41$, SiMe_3], 13.07 [$^1J(^{13}\text{C}-^{117/119}\text{Sn}) = 959/1004$, CH_2], 24.33 (O_2CMe), 178.51 [$^2J(^{13}\text{C}-^{117/119}\text{Sn}) = 32$, $\text{C}=\text{O}$]; $^{119}\text{Sn}\{^1\text{H}\}$ NMR (100.73 MHz, CDCl_3): δ -477.8 [$^2J(^{119}\text{Sn}-\text{O}-^{117}\text{Sn}) = 249$, $^2J(^{119}\text{Sn}-\text{O}-^{117}\text{Sn}) = 123$]. Analysis calculated for $\text{C}_{36}\text{H}_{84}\text{O}_{18}\text{Si}_6\text{Sn}_6$: C 25.65, H 5.02%; found: C 25.43, H 4.92%.

Crystal data

$[\text{Sn}_6\text{O}_6(\text{C}_2\text{H}_3\text{O}_2)_6(\text{C}_4\text{H}_{11}\text{Si})_6]$
 $M_r = 1685.71$
 Monoclinic, $P2_1/n$
 $a = 13.8888$ (7) Å
 $b = 18.8359$ (9) Å
 $c = 13.9301$ (7) Å
 $\beta = 116.545$ (1)°
 $V = 3260.1$ (3) Å³
 $Z = 2$

$D_x = 1.717$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 4788 reflections
 $\theta = 2.7$ – 29.4°
 $\mu = 2.43$ mm⁻¹
 $T = 223$ (2) K
 Plate, colourless
 $0.23 \times 0.18 \times 0.07$ mm

Data collection

Bruker AXS SMART CCD diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 2000)
 $T_{\min} = 0.549$, $T_{\max} = 0.844$
 27420 measured reflections

9482 independent reflections
 7215 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.038$
 $\theta_{\max} = 30.1^\circ$
 $h = -17 \rightarrow 19$
 $k = -23 \rightarrow 26$
 $l = -19 \rightarrow 19$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.093$
 $S = 1.02$
 9482 reflections
 301 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.04P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 1.12$ e Å⁻³
 $\Delta\rho_{\min} = -0.71$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Sn1—O1	2.078 (2)	Sn2—C11	2.113 (4)
Sn1—O2	2.079 (3)	Sn3—O1 ⁱ	2.075 (2)
Sn1—O3	2.080 (2)	Sn3—O2	2.066 (6)
Sn1—O4	2.157 (3)	Sn3—O3	2.099 (3)
Sn1—O6	2.150 (2)	Sn3—O7	2.152 (3)
Sn1—C7	2.117 (4)	Sn3—O9 ⁱ	2.149 (2)
Sn2—O1	2.085 (3)	Sn3—C15	2.114 (4)
Sn2—O2	2.085 (2)	Sn1—Sn2	3.2036 (4)
Sn2—O3 ⁱ	2.075 (2)	Sn1—Sn3	3.2007 (4)
Sn2—O5	2.153 (3)	Sn2—Sn3 ⁱ	3.2020 (4)
Sn2—O8	2.162 (3)		
O1—Sn1—O6	158.61 (10)	O3 ⁱ —Sn2—O5	158.49 (10)
O2—Sn1—C7	178.06 (12)	O1 ⁱ —Sn3—O7	158.53 (10)
O3—Sn1—O4	162.12 (10)	O2—Sn3—O9 ⁱ	157.81 (10)
O1—Sn2—C11	175.60 (12)	O3—Sn3—C15	175.13 (13)
O2—Sn2—O8	159.53 (10)		

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

The C-bound H atoms were included in the riding-model approximation, with C—H distances for methylene groups of 0.98 Å and C—H = 0.97 Å for methyl; $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{CH}_2)$ and $U_{\text{iso}}(\text{methyl H}) = 1.5U_{\text{eq}}(\text{CH}_3)$. The largest residual electron density peak is located in the vicinity of the atom Sn2.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *PATY* in *DIRDIF92* (Beurskens *et al.*, 1992); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

The Australian Research Council and the National University of Singapore (R-143-000-213-112) are thanked for support.

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