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cis-1,1,2,2,3,4,4,5,5,6-Decamethyl-1,2,4,5-tetrastannacyclohexane, $C_{12}H_{32}Sn_4$

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Abstract. $M_r = 651\cdot1$, monoclinic, $P2_1/n$, $a = 10\cdot650$ (6), $b = 12\cdot313$ (6), $c = 16\cdot409$ (6) Å, $\beta = 95\cdot27$ (5)°, $U = 2125$ (3) Å^3, $Z = 4$, $D_x = 2\cdot04$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 4\cdot7$ mm^{-1}, $F(000) = 1216$, $T = 294$ (1) K, final $R = 0\cdot030$ for 2057 unique diffractometer data ($F_o > 3\sigma$) and 148 refined parameters. The molecule is in the boat conformation. The Sn—Sn bond lengths are 2·775 (2) and 2·766 (2) Å.

Introduction. Although the structures of cyclohexane and its derivatives are well documented, relatively little is known about the structures of metallacyclohexanes. We have recently prepared a series of tetrastannacyclohexanes with methyl groups attached to tin (Mitchell & Fabisch, 1983), while Jurkschat & Gielen (1982) have described the preparation of a tetrastannacyclohexane with phenyl groups bonded to tin.

NMR spectroscopic studies indicate a possible change in ring geometry on replacing methylene protons on the ring carbons by methyl groups. In order to get more information regarding possible changes of ring conformation on the introduction of different substituents we decided to determine the crystal structure of 1,1,2,2,3,4,4,5,5,6-decamethyl-1,2,4,5-tetrastannacyclohexane; this is a particularly interesting molecule since it can in principle exist as the *cis* or *trans* isomer. However, its preparation leads to the formation of only one molecular species; ^{13}C NMR shows that the methyl groups on tin are non-equivalent, in contrast to the situation for the corresponding octa- and dodecamethyltetrastannacyclohexanes, which show only one methyltin resonance.

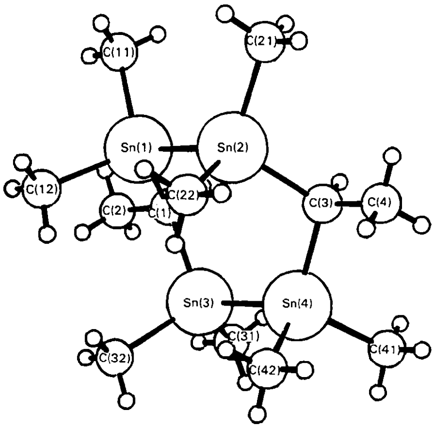

Fig. 1. General view of the molecule.

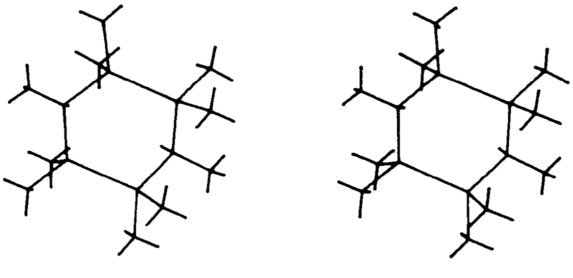

Fig. 2. Stereoscopic view of the molecule.

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Experimental. Prepared by treating 1,3-dibromo-1,1,2,3,3-pentamethyl-1,3-distannapropane with sodium in liquid ammonia; colourless crystals obtained by recrystallization from CH_2Cl_2/CH_3OH, m.p. 352–353 K, yield 68%; $\omega/2\theta$ scan, scan speed 0·95–2·5° min^{-1} in θ, Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$, crystal 0·10 × 0·22 × 0·21 mm, lattice parameters from least-squares fit with 25 reflexions in the range $10{\cdot}4 \leq \theta \leq 14{\cdot}2°$; three standard reflexions recorded every 5 h show a decrease up to 15%; 4110 reflexions measured, $2 \leq \theta \leq 25°$, $\pm h$, $+k$, $+l$ (h 0–12, k 0–14, l 0–19); Lorentz–polarization and decay corrections, absorption correction *via* ψ scans, max./min. transmission 1·0/0·61; structure solution with *MULTAN*82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) (Sn positions), ΔF syntheses and full-matrix least squares with 2057 observed reflexions $[F_o > 3\sigma(F_o)]$ and anisotropic temperature factors for Sn and C atoms; H atoms placed in geometrically calculated positions (C—H 0·95 Å), only two common isotropic temperature factors for H atoms refined, complex neutral-atom scattering factors from Cromer & Waber (1974) and Cromer (1974) (H: SDS, C: RHF), least-squares refinement based on $|F|$ and 148 refined parameters, extinction correction $[g = 1{\cdot}53\,(9) \times 10^{-8}]$ (Stout & Jensen, 1968), $R = 0{\cdot}030$, $R_w = 0{\cdot}031$, unit weights, maximum Δ/σ 0·03, no significant features in final ΔF synthesis; programs: Enraf–Nonius structure determination package (Frenz, 1981), *ORTEP* (Johnson, 1976), *POP*1 (van de Waal, 1976).

Discussion. The structure of the title compound is shown in Fig. 1 (*ORTEP*) and in a stereoview in Fig. 2 (*POP*1). Positional parameters and the equivalent values of the anisotropic temperature factors β_{ij} are given in Table 1.* Bond lengths and angles are given in Table 2. The asymmetric unit of the crystal contains one molecule which shows no crystallographic symmetry but nearly *mm*2 symmetry.

The most interesting feature of this study is that the title compound exists in the crystalline state as the *cis* isomer in the boat conformation, whereas the closely related 2,2,3,3,5,5,6,6-octaphenyl-2,3,5,6-tetrastannacyclohexane ($C_{50}H_{44}Sn_4$) has a chair structure (Meunier-Piret, Van Meerssche, Gielen & Jurkschat, 1983). The Sn—Sn bond lengths in the title compound are in the range of the Sn—Sn bond lengths in $(C_6H_5)_6Sn_2$ (Preut, Haupt & Huber, 1973): 2·780 (4), 2·759 (4) Å, $[(C_6H_5)_2Sn]_6$ (Olson & Rundle, 1963): 2·77, 2·78, 2·78 Å and in $C_{50}H_{44}Sn_4$ (Meunier-Piret, Van Meerssche, Gielen & Jurkschat, 1983): 2·783 (1) Å and are markedly shorter than those in $[\{(CH_3)_2SiCH_2\}_2Sn]_4$ (Belsky, Zemlyansky, Kolosova & Borisova, 1981): 2·834 (1), 2·829 (1) Å and in Ar_6Sn_3(Ar = 2,6-diethylphenyl) (Masamune, Sita & Williams, 1983): 2·870 (1), 2·856 (1), 2·854 (1) Å. The Sn—C bond lengths are in the normal range and the maximum deviation of the C—Sn—C, C—Sn—Sn and Sn—C—Sn angles from the ideal tetrahedral angle is 3·8 (4)°, whereas in $C_{50}H_{44}Sn_4$ the mean value of the angles Sn—C—Sn is much more distorted (120·6°) from the tetrahedral angle.

Intermolecular distances do not indicate the existence of interactions exceeding van der Waals forces.

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Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å²)

$$B_{eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Sn(1)	0·03213 (5)	0·78410 (5)	0·30744 (4)	3·56 (1)
Sn(2)	−0·21053 (5)	0·77216 (5)	0·35904 (4)	3·42 (1)
Sn(3)	−0·10494 (5)	0·77805 (5)	0·10004 (4)	3·46 (1)
Sn(4)	−0·34753 (5)	0·77414 (5)	0·15083 (3)	3·34 (1)
C(1)	0·0303 (8)	0·8556 (7)	0·1882 (6)	3·8 (2)
C(2)	0·1641 (9)	0·8574 (9)	0·1582 (6)	5·9 (3)
C(3)	−0·3557 (7)	0·8402 (7)	0·2720 (5)	3·3 (2)
C(4)	−0·4874 (8)	0·8262 (9)	0·3017 (6)	4·9 (2)
C(11)	0·1604 (9)	0·8759 (9)	0·3901 (6)	6·5 (3)
C(12)	0·113 (1)	0·6243 (8)	0·3016 (6)	5·7 (3)
C(21)	−0·225 (1)	0·8555 (9)	0·4729 (6)	5·9 (3)
C(22)	−0·2604 (9)	0·6054 (8)	0·3787 (6)	5·3 (3)
C(31)	−0·101 (1)	0·8617 (9)	−0·0147 (6)	5·7 (3)
C(32)	−0·043 (1)	0·6133 (8)	0·0820 (7)	6·1 (3)
C(41)	−0·416 (1)	0·6087 (8)	0·1543 (7)	6·4 (3)
C(42)	−0·4825 (9)	0·8607 (8)	0·0684 (6)	5·5 (3)

Table 2. *Bond lengths* (Å) *and angles* (°)

Sn(1)—Sn(2)	2·775 (2)	Sn(3)—Sn(4)	2·766 (2)
Sn(1)—C(1)	2·14 (1)	Sn(3)—C(1)	2·16 (1)
Sn(1)—C(11)	2·15 (1)	Sn(3)—C(31)	2·15 (1)
Sn(1)—C(12)	2·15 (1)	Sn(3)—C(32)	2·16 (1)
Sn(2)—C(21)	2·15 (1)	Sn(4)—C(41)	2·16 (1)
Sn(2)—C(22)	2·15 (1)	Sn(4)—C(42)	2·15 (1)
Sn(2)—C(3)	2·17 (1)	Sn(4)—C(3)	2·16 (1)
C(1)—C(2)	1·54 (1)	C(3)—C(4)	1·52 (1)
C(1)—Sn(1)—C(11)	108·2 (4)	C(1)—Sn(3)—C(31)	108·0 (4)
C(1)—Sn(1)—C(12)	107·8 (4)	C(1)—Sn(3)—C(32)	108·6 (4)
C(1)—Sn(1)—Sn(2)	111·7 (2)	C(1)—Sn(3)—Sn(4)	112·3 (2)
C(11)—Sn(1)—C(12)	106·2 (4)	C(31)—Sn(3)—C(32)	107·4 (4)
C(11)—Sn(1)—Sn(2)	112·4 (3)	C(31)—Sn(3)—Sn(4)	111·5 (3)
C(12)—Sn(1)—Sn(2)	110·3 (3)	C(32)—Sn(3)—Sn(4)	108·9 (3)
Sn(1)—Sn(2)—C(21)	112·3 (3)	Sn(3)—Sn(4)—C(41)	110·1 (3)
Sn(1)—Sn(2)—C(22)	110·0 (2)	Sn(3)—Sn(4)—C(42)	112·3 (2)
Sn(1)—Sn(2)—C(3)	113·3 (2)	Sn(3)—Sn(4)—C(3)	112·8 (2)
C(21)—Sn(2)—C(22)	106·7 (4)	C(41)—Sn(4)—C(42)	106·2 (4)
C(21)—Sn(2)—C(3)	106·7 (3)	C(41)—Sn(4)—C(3)	106·8 (4)
C(22)—Sn(2)—C(3)	107·5 (3)	C(42)—Sn(4)—C(3)	108·2 (3)
Sn(1)—C(1)—Sn(3)	112·3 (4)	Sn(2)—C(3)—Sn(4)	112·1 (3)
Sn(1)—C(1)—C(2)	111·5 (6)	Sn(2)—C(3)—C(4)	110·9 (6)
C(2)—C(1)—Sn(3)	111·2 (6)	C(4)—C(3)—Sn(4)	111·5 (5)

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39002 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Tetrakis[μ_3-(4-methoxy-2-butyl)-*O*,μ_3-*C*^{2}]tetralithium, [$Li_4(C_5H_{11}O)_4$]

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Abstract. $M_r = 376{\cdot}33$, tetragonal, $P\bar{4}b2$, $a = 12{\cdot}389$ (3), $c = 8{\cdot}218$ (1) Å, $U = 1261{\cdot}4$ (5) Å^3, $Z = 2$, $D_x = 0{\cdot}991$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, μ(Cu $K\alpha$) = 4·3 cm^{-1}, $F(000) = 416$, $T = 295$ K, $R = 0{\cdot}046$ for 664 observed reflections with $I > 2{\cdot}5\sigma(I)$. 3-Lithio-1-methoxybutane crystallizes from pentane as chelated *meso* tetramers with $\bar{4}$ site symmetry. The four Li atoms form a slightly distorted tetrahedron.

Introduction. Any attempt to understand the enhanced reactivity exhibited by Lewis-base adducts of alkyllithium tetramers in certain reactions – Wurtz coupling (Eastham & Gibson, 1963), iodine–lithium exchange (Applequist & O'Brien, 1963), bromine–lithium exchange (Rogers & Houk, 1982), ethylenation reaction (Bartlett, Goebel & Weber, 1969) – requires the knowledge of structure and bonding in these species. So far ^{7}Li NMR has revealed the tetrahedral nature of alkyllithiums dissolved in diethyl ether (Brown, 1970; McKeever, 1972). More detailed information about the structure of alkyllithium etherates has remained unavailable due to the failure to obtain crystalline materials from these solutions. We now report the crystal structure of the intramolecularly etherated title compound that was obtained from the corresponding Hg derivative by reaction with Li in pentane (Geurink, 1982). A preliminary discussion of the results has been given previously (Klumpp, Geurink, Spek & Duisenberg, 1983).

Experimental. Enraf–Nonius CAD-4F diffractometer, block-shaped (~0·5 × 0·5 × 0·5 mm) transparent colourless crystal mounted under vacuum in a Lindemann-glass capillary [$\theta < 70°$, $\omega/2\theta$ scan, $+h$, $+k$, $+l$: $k > h$; Cu $K\alpha$ (Ni-filtered)]. 868 reflections collected and corrected for Lorentz–polarization effects and for long- and short-term instabilities as monitored by four reference reflections (322, $\bar{3}2\bar{2}$, $\bar{3}\bar{2}2$, $3\bar{2}\bar{2}$): max. correction 2%, no indication of decay, 752 unique reflections. The observed extinctions for $0kl$ ($k = 2n + 1$) did not fix the space group uniquely. The correct symmetry was deduced *via* the successful structure determination and refinement in space group $P\bar{4}b2$. Solution by direct methods, refinement by full-matrix least-squares techniques on F: all non-hydrogen atoms with anisotropic thermal parameters and the H atoms with one overall isotropic thermal parameter, and all positional parameters; H-atom positions from a difference Fourier map. Final $R = 0{\cdot}0457$, $wR = 0{\cdot}0459$ [$w = 1$, 664 reflections with $I > 2{\cdot}5\sigma(I)$]; $(\Delta/\sigma)_{\max} = 0{\cdot}4$; 74 parameters; minimum and maximum residual densities $-0{\cdot}11$, $0{\cdot}13$ e Å^{-3}.

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