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Structure of 2,2-Bis[3-(dimethylamino)propyl]-1,1,1,3,3,3-hexaphenyltristannane-(2Sn–Sn)

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Abstract. $[\text{Sn}_3(\text{C}_6\text{H}_5)_6(\text{C}_5\text{H}_{12}\text{N})_2]$, $\text{C}_{46}\text{H}_{54}\text{N}_2\text{Sn}_3$, $M_r = 991.1$, orthorhombic, *Pbca*, $a = 16.835$ (5), $b = 21.834$ (7), $c = 24.720$ (8) Å, $V = 9086$ (4) Å³, $Z = 8$, $D_x = 1.45$ g cm⁻³, $\lambda(\text{Mo K}\alpha) = 0.71069$ Å, $\mu = 16.8$ cm⁻¹, $F(000) = 3952$, $T = 293$ K, final $R = 0.050$ ($wR = 0.051$) for 3392 observed reflections. The Sn–Sn distances are 2.772 (1) and 2.777 (1) Å and the Sn1–Sn2–Sn3 angle is 111.31 (3)°. There are no intra- or intermolecular Sn–N interactions; all three tin atoms are tetrahedrally coordinated.

Introduction. Since the synthesis of $[(\text{Me}_3\text{Si})_2\text{CH}]_2\text{Sn}$ as the first stable carbon bonded stannylene with coordination number two (Davidson & Lappert, 1973) the chemistry of this class of compounds has attracted more and more interest. In recent papers it has been shown that not only bulky substituents stabilize monomeric tin(II) compounds but also intramolecular donor–acceptor interactions or a combination of both (Angermund, Jonas, Krüger, Latten & Tsay, 1988; Jastrzebski, van der Schaaf, Boersma, van Koten, Zoutberg & Heijdenrijk, 1989; Engelhardt, Jolly, Lappert, Raston & White, 1988; Jurkschat, Klaus, Dargatz, Tzschach, Meunier-Piret & Mahieu, 1989). Within our studies on this subject (Jurkschat *et al.*, 1989) we have prepared $(\text{Me}_2\text{NCH}_2\text{CH}_2\text{CH}_2)_2\text{SnCl}_2$ (1) and performed its reduction with sodium/naphthalene. However, this

reaction did not give, as might be expected, the monomeric stannylene $(\text{Me}_2\text{NCH}_2\text{CH}_2\text{CH}_2)_2\text{Sn}$ (2) but only a mixture of oligomers which have not been isolated and identified. Neumann and co-workers have shown that stannylenes $R_2\text{Sn}$ can also be generated by thermolysis or photolysis of polystannanes (Neumann & Schwarz, 1975). However, analogous investigations on the title compound $(\text{Me}_2\text{NCH}_2\text{CH}_2\text{CH}_2)_2\text{Sn}(\text{SnPh}_3)_2$ (3) did not provide evidence for a stable stannylene (2) or related definite oligomers (Klaus & Jurkschat, 1988). To our knowledge, (3) is only the second known oligostannane containing a functional substituent in the form of the dimethylaminopropyl group (*cf.* the survey of literature referring to this given by Adams & Dräger, 1988). We report here the X-ray analysis of (3) which does not indicate any intramolecular Sn–N interaction. This might explain the above mentioned failure of (3) to give the stannylene (1) by thermolysis or photolysis.

Experimental. The title compound (3) was prepared by reaction of $(\text{Me}_2\text{NCH}_2\text{CH}_2\text{CH}_2)_2\text{Sn}(\text{NEt}_2)_2$ with Ph_3SnH (Klaus & Jurkschat, 1988). The colorless crystals obtained by recrystallization from toluene decomposed slowly by X-ray treatment; therefore, the crystal selected for the diffraction experiments with dimensions $0.28 \times 0.39 \times 0.41$ mm was covered with a thin protective film of shellac and then mounted on a Huber four-circle diffractometer; graphite-

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Table 1. Positional parameters and equivalent isotropic displacement parameters (\AA^2) of non-H atoms, with e.s.d.'s in parentheses

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	x	y	z	U_{eq}
Sn1	0.03062 (4)	0.11145 (3)	0.16649 (3)	0.0785 (3)
Sn2	0.13505 (4)	0.17827 (3)	0.23035 (3)	0.0767 (3)
Sn3	0.23971 (4)	0.24578 (4)	0.16756 (3)	0.0774 (3)
C1	-0.0494 (6)	0.0583 (5)	0.2161 (4)	0.078 (4)
C2	-0.0740 (7)	0.0816 (5)	0.2656 (5)	0.097 (5)
C3	-0.1237 (8)	0.0485 (6)	0.2976 (6)	0.110 (6)
C4	-0.1485 (8)	-0.0078 (7)	0.2819 (6)	0.124 (7)
C5	-0.1263 (8)	-0.0319 (5)	0.2350 (6)	0.119 (6)
C6	-0.0749 (8)	0.0004 (6)	0.2001 (5)	0.108 (6)
C7	-0.0403 (6)	0.1676 (5)	0.1152 (4)	0.078 (4)
C8	-0.1204 (8)	0.1559 (6)	0.1095 (5)	0.114 (6)
C9	-0.1672 (10)	0.1902 (7)	0.0768 (7)	0.148 (9)
C10	-0.1412 (10)	0.2402 (9)	0.0488 (6)	0.143 (8)
C11	-0.0613 (9)	0.2533 (6)	0.0532 (5)	0.118 (6)
C12	-0.0124 (7)	0.2176 (6)	0.0870 (5)	0.103 (5)
C13	0.0900 (7)	0.0496 (6)	0.1125 (5)	0.093 (5)
C14	0.1528 (8)	0.0148 (6)	0.1279 (6)	0.109 (6)
C15	0.1922 (10)	-0.0249 (7)	0.0943 (7)	0.128 (7)
C16	0.1670 (9)	-0.0311 (7)	0.0429 (8)	0.138 (8)
C17	0.1074 (9)	0.0059 (8)	0.0247 (6)	0.152 (8)
C18	0.0684 (8)	0.0466 (6)	0.0592 (6)	0.130 (7)
C19	0.1780 (6)	0.3102 (5)	0.1162 (4)	0.077 (4)
C20	0.1232 (8)	0.3508 (6)	0.1368 (5)	0.112 (6)
C21	0.0845 (8)	0.3937 (6)	0.1062 (6)	0.114 (6)
C22	0.0987 (8)	0.3942 (7)	0.0530 (6)	0.125 (7)
C23	0.1520 (8)	0.3574 (7)	0.0305 (5)	0.118 (7)
C24	0.1931 (7)	0.3138 (6)	0.0618 (5)	0.101 (5)
C25	0.3111 (7)	0.1903 (5)	0.1154 (4)	0.080 (5)
C26	0.2744 (8)	0.1484 (5)	0.0845 (6)	0.106 (6)
C27	0.3180 (10)	0.1084 (7)	0.0505 (6)	0.126 (7)
C28	0.3970 (10)	0.1143 (7)	0.0482 (5)	0.115 (7)
C29	0.4357 (7)	0.1578 (7)	0.0790 (6)	0.108 (6)
C30	0.3915 (7)	0.1974 (6)	0.1115 (5)	0.095 (5)
C31	0.3171 (7)	0.2983 (5)	0.2169 (4)	0.085 (5)
C32	0.3472 (7)	0.2766 (6)	0.2656 (5)	0.100 (5)
C33	0.3991 (10)	0.3119 (8)	0.2973 (6)	0.141 (8)
C34	0.4185 (10)	0.3681 (9)	0.2801 (7)	0.145 (9)
C35	0.3918 (10)	0.3925 (7)	0.2336 (8)	0.147 (8)
C36	0.3404 (8)	0.3554 (7)	0.2016 (6)	0.122 (7)
C37	0.1998 (6)	0.1152 (5)	0.2808 (4)	0.104 (5)
C38	0.1453 (7)	0.0711 (6)	0.3118 (6)	0.144 (7)
C39	0.1924 (7)	0.0279 (7)	0.3481 (6)	0.169 (8)
N1	0.1389 (8)	-0.0146 (6)	0.3780 (4)	0.188 (9)
C40	0.1976 (8)	-0.0574 (6)	0.4018 (4)	0.372 (18)
C41	0.0940 (8)	0.0179 (6)	0.4136 (4)	0.273 (17)
C42	0.0729 (6)	0.2388 (5)	0.2848 (4)	0.096 (6)
C43	0.1284 (7)	0.2826 (6)	0.3147 (6)	0.138 (7)
C44	0.0866 (9)	0.3288 (6)	0.3514 (6)	0.165 (9)
N2	0.0582 (8)	0.3007 (5)	0.4016 (6)	0.181 (9)
C45	0.0038 (8)	0.3503 (5)	0.4250 (6)	0.261 (10)
C46	0.1351 (8)	0.3002 (5)	0.4315 (6)	0.319 (18)

were 'riding' on the corresponding C atoms during refinement. $\sum w(\Delta F)^2$ minimized; Final weighting scheme was $w^{-1} = \sigma^2 + 5.3 \times 10^{-5} F^2$; an empirical extinction correction was applied with $F_{\text{corr}} = F_c(1 - 10^{-9} \chi F_c / \sin \theta)$ where χ refined to 3.0 (2). Final R was 0.050 ($wR = 0.051$); $S = 3.57$; 7.4 reflections/parameter; in final cycle $\Delta/\sigma \leq 0.17$; max. and min. heights in final difference Fourier map 0.51 and -0.33 e \AA^{-3} , respectively; atomic scattering factors from *SHELX76* (Sheldrick, 1976). Calculations were performed on IBM compatible PC's using the program packages *SHELX76* (Sheldrick, 1976), *SHELXS86* (Sheldrick, 1986) and *EDIT* (Jaskólski, 1982).

Discussion. Final fractional coordinates and equivalent isotropic displacement parameters of (3) are listed in Table 1.* The molecular structure is shown in Fig. 1; selected bond lengths, bond angles and torsion angles are given in Table 2.

All three Sn atoms have the coordination number four and a very slightly distorted tetrahedral environment [the maximum deviation from the ideal geometry is $4.9 (4)^\circ$ for C7—Sn1—C13]. There is no indication of any Sn—N interactions. The non-formation of a donor—acceptor bond between nitrogen and tin might be caused by both steric hindrance and the limited Lewis acidity of the Sn atoms. The two Sn—Sn bond lengths are the same. They agree very well with the Sn—Sn distance in $(\text{Ph}_3\text{Sn})_2$

* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles, torsion angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54272 (65 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

monochromated Mo $K\alpha$ radiation was used. Lattice parameters were derived from the least-squares refinement of the setting angles of 15 reflections in the range $12 < \theta < 15^\circ$. θ - 2θ scan; $2\theta_{\text{max}} = 58^\circ$; h, k, l range from 0,0,0 to 22,29,33; 6978 unique reflections, 3392 with $|F| \geq 4.00\sigma(F)$ considered observed. During data collection the intensities of check reflections 624 and 264 decreased by 14.0% of their starting values. The decrease, obviously caused by slight decomposition, was compensated for by a linear decay correction. Lp correction was carried out but absorption effects were ignored. The structure was solved by Patterson methods and subsequent Fourier syntheses and refined on F by full-matrix least-squares procedures with anisotropic displacement parameters for the non-H atoms. H-atom positions were calculated geometrically and

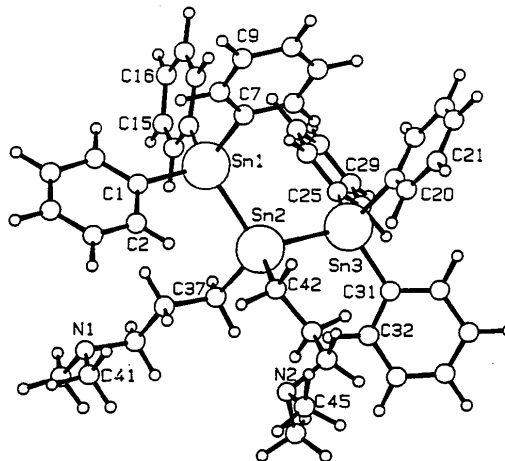


Fig. 1. Molecular structure of $(\text{Me}_2\text{NCH}_2\text{CH}_2)_2\text{Sn}(\text{SnPh}_3)_2$ with atom-numbering scheme (some C atoms are unnumbered for the sake of clarity but can be unequivocally identified).

Table 2. Selected bond lengths (Å), bond angles (°) and torsion angles (°), with e.s.d.'s in parentheses

Sn1—Sn2	2.777 (1)	Range of Sn—C:	2.12 (1)–2.16 (1)
Sn2—Sn3	2.772 (1)		
C1—Sn1—C7	107.3 (4)	C37—Sn2—C42	106.0 (4)
C1—Sn1—C13	107.8 (4)	C37—Sn2—Sn3	110.0 (3)
C7—Sn1—C13	104.6 (4)	C42—Sn2—Sn3	109.4 (3)
C1—Sn1—Sn2	110.8 (3)	Sn2—Sn3—C19	111.7 (3)
C7—Sn1—Sn2	113.0 (3)	Sn2—Sn3—C25	113.2 (3)
C13—Sn1—Sn2	112.9 (3)	Sn2—Sn3—C31	110.8 (3)
Sn1—Sn2—Sn3	111.31 (3)	C19—Sn3—C25	106.5 (4)
Sn1—Sn2—C37	108.3 (3)	C19—Sn3—C31	106.4 (4)
Sn1—Sn2—C42	111.7 (3)	C25—Sn3—C31	107.9 (4)
C1—Sn1—Sn2—Sn3	179.3 (3)	Sn1—Sn2—Sn3—C25	−60.0 (3)
C7—Sn1—Sn2—Sn3	−60.3 (2)	Sn1—Sn2—Sn3—C31	178.6 (3)
C13—Sn1—Sn2—Sn3	58.2 (3)	C37—C38—C39—N1	180 (1)
Sn1—Sn2—Sn3—C19	60.2 (3)	C42—C43—C44—N2	76 (1)

[2.770 (1) Å; Preut, Haupt & Huber, 1973] and are only slightly shorter than those in *tert*-Bu₂Sn(SnPh₃)₂ [2.801, 2.795 Å; Adams & Dräger, 1987] and lie in the range generally observed for polytin species without special steric demands of the substituents (2.75–2.83 Å; Puff *et al.*, 1989). The Sn—C distances do not differ significantly [average 2.14 (3) Å] and are quite normal.

With respect to the Sn1—Sn2 and Sn2—Sn3 bonds, respectively, the six substituents at both tin atoms adopt in each case a nearly perfect staggered conformation. This is in agreement with the general existence of an all-*trans* conformation of the Sn_n units observed for a series of linear organo-substituted polystannanes (Adams & Dräger, 1987). The three phenyl rings attached to one Sn atom are twisted in a propeller-like manner. Looking from Sn1 to Sn3, the six phenyl rings are perfectly eclipsed with regard to their Sn—C bonds. Surprisingly, the dimethylaminopropyl groups exhibit different conformations. As can be seen from Fig. 1 and Table 2,

the chain C37—N1 adopts an extended all-*trans* conformation whereas in the chain C42—N2 a *gauche* conformation is adopted.

Within the crystal lattice the molecules are separated by normal van der Waals distances.

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Structure of Methylammonium Hydrogen Malonate (I) and Methylammonium Malonate (II)

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Abstract. (I), CH₆N⁺.C₃H₃O₄[−], *M_r* = 135.12, monoclinic, *P*2₁/*c*, *a* = 8.017 (1), *b* = 9.245 (1), *c* =

8.582 (1) Å, β = 92.78 (1)°, *V* = 635.3 (2) Å³, *T* = 293 (2) K, *D_m* = 1.39 (2), *D_x* = 1.413 g cm^{−3}, *Z* = 4, *F*(000) = 288, Mo *K*α radiation, λ = 0.71069 Å, μ = 1.187 cm^{−1}, final *R* = 0.033 and *wR* = 0.031 for 1191 observed reflections. (II), 2CH₆N⁺.C₃H₂O₄^{2−}, *M_r* = 166.18, monoclinic, *P*2₁/*c*, *a* = 9.185 (1), *b* =

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