and $(NH_4)_2CuCl_3$ salt structures. Constrained by the inversion center symmetry, the distortion of the $[Cu_2Cl_6]^{2-}$ dimer, which is crucial for magnetic behavior of this complex, is of a twisting mode, *i.e.* the plane defined by a Cu atom and two terminal Cl atoms, Cu/Cl(1)/Cl(3), is twisted by an angle of 45° with respect to the Cu—Cl(2)—Cu—Cl(2) plane.

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1,1,2,2,4,4,5,5-Octamethyl-1,2,4,5-tetrastannacyclohexane

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Abstract. $C_{10}H_{28}Sn_4$, $M_r = 623{\cdot}09$, monoclinic, $P2_1/c$, $a = 20{\cdot}095$ (6), $b = 6{\cdot}492$ (2), $c = 16{\cdot}067$ (7) Å, $\beta = 112{\cdot}88$ (3)°, $V = 1931$ (1) Å^3, $Z = 4$, $D_x = 2{\cdot}143$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 5{\cdot}12$ mm^{-1}, $F(000) = 1152$, $T = 291$ (1) K, final $R = 0{\cdot}070$ for 1243 unique observed $[F \geq 4{\cdot}0\sigma(F)]$ diffractometer data. The molecule is in the boat conformation. The Sn—Sn bond lengths are 2·791 (3) and 2·780 (3) Å; this small difference, which is greater than three times the corresponding standard deviation, parallels the two different values for the one-bond Sn–Sn coupling constant observed in the ^{119}Sn solid-state spectrum of the compound.

Introduction. Previous work (Meunier-Piret, Van Meerssche, Gielen & Jurkschat, 1983; Preut, Bleckmann, Mitchell & Fabisch, 1984) has shown that 1,2,4,5-tetrastannacyclohexanes can exist in both chair and boat forms: the chair form is observed for the octaphenyl derivative, while the *cis*-dodecamethyl derivative exists in a boat structure. In order to obtain more information on the factors determining which structure is preferred, we decided to determine the crystal structure of 1,1,2,2,4,4,5,5-octamethyl-1,2,4,5-tetrastannacyclohexane. The high-resolution solid-state ^{119}Sn NMR spectrum of this compound reveals four non-equivalent Sn signals (Harris, Mitchell & Nesbitt, 1985) with two different one-bond Sn–Sn coupling constants (Mitchell, 1986): these may be related to differences in the Sn—Sn bond length.

Experimental. Title compound prepared by treating 1,3-dibromo-1,1,3,3-tetramethyl-1,3-distannapropane with sodium in liquid ammonia; colourless crystals obtained by vacuum sublimation, m.p. 372–373 K, yield 73% (Mitchell, Fabisch, Wickenkamp, Kuivila & Karol, 1986). Crystal size 0·06 × 0·06 × 0·38 mm. The external form of the crystals and the X-ray photographs indicated poor quality of the crystals which diffracted weakly. $\omega/2\theta$ scan, scan speed 2·5–14·6° min^{-1} in θ, Nicolet $R3m/V$ diffractometer, graphite-monochromated Mo $K\alpha$; lattice parameters from least-squares fit with 22 reflections up to $2\theta = 24{\cdot}3°$; six standard reflections recorded every 2·5 h showed up to 52% intensity loss; 4773 reflections measured, $1{\cdot}5 \leq \theta \leq 21{\cdot}0°$, $-17 \leq h \leq 17$, $0 \leq k \leq 7$, $-21 \leq l \leq 21$; after averaging ($R_{int} = 0{\cdot}061$): 2099 unique reflections, 1243 with $F \geq 4{\cdot}0\sigma(F)$; Lorentz–polarization correction, decay correction, no absorption correction; systematic absences $(h0l)$ $l = 2n + 1$, $(0k0)$ $k = 2n + 1$ conform to space group $P2_1/c$; structure solution *via* Patterson function, ΔF syntheses and full-matrix least-squares refinement with anisotropic temperature factors for all non-H atoms and a common isotropic temperature factor for H atoms, which were placed in geometrically calculated positions (C—H 0·96 Å); refinement on F with 1243 reflections and 128 refined parameters; $w = 1{\cdot}0/[\sigma^2(F) + 0{\cdot}0005F^2]$; $S = 1{\cdot}62$, $R = 0{\cdot}070$, $wR = 0{\cdot}060$, $(\Delta/\sigma)_{max} = 0{\cdot}30$, no extinction correction; largest peak in final ΔF map $\pm 1{\cdot}4$ (6) e Å^{-3}, atomic

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scattering factors for neutral atoms and real and imaginary dispersion terms from *International Tables for X-ray Crystallography* (1974); programs: *PARST* (Nardelli, 1983), *SHELXTL-Plus* (Sheldrick, 1987). The molecule and the numbering scheme are shown in Fig. 1, packing in Fig. 2. Positional parameters and the equivalent values of the anisotropic temperature factors for the non-H atoms are given in Table 1.* Bond lengths and angles are given in Table 2.

Discussion. The molecule studied here exists in the crystalline state in a boat conformation, while the corresponding octaphenyl derivative adopts a chair form (Meunier-Piret *et al.*, 1983): thus the two additional methyl groups in the *cis*-decamethyl derivative are clearly not responsible for the transition to a boat form (Preut *et al.*, 1984): it appears at present that the nature of the groups attached to Sn in R_2Sn moieties will determine the preferred geometry. The Sn—Sn bond lengths in the title compound are noticeably longer than in the decamethyl derivative [2·775 (2) and 2·766 (2) Å], while the C—Sn—Sn and Sn—C—Sn angles are smaller [average values for the decamethyl compound: 111·5 (3) and 112·2 (4)° respectively]. However, the Sn—C—Sn angle in the octaphenyl derivative [120·6 (1)°] is considerably more distorted from the tetrahedral angle. The NMR results noted in the *Introduction* agree with the present crystal structure and indicate that the small Sn—Sn bond-length difference, which is greater than three times the corresponding standard deviation, is indeed reflected by differences in the one-bond Sn–Sn coupling constant: further studies will hopefully shed more light on this question.

* Lists of H-atom coordinates, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51322 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

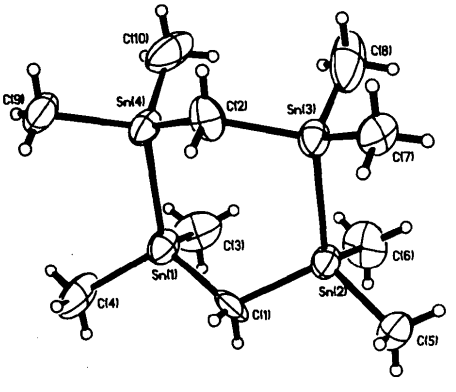

Fig. 1. General view (*SHELXTL-Plus* graphic) of the molecule, showing the atom-numbering scheme. The ellipsoids correspond to 50% probability.

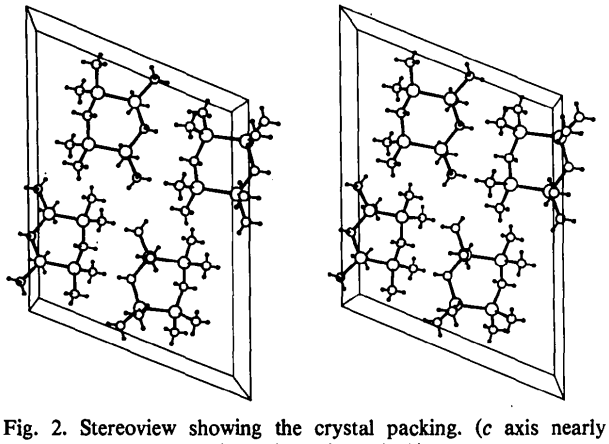

Fig. 2. Stereoview showing the crystal packing. (*c* axis nearly horizontal, *a* axis vertical.)

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2 × 10^3)

$$U_{eq} = \tfrac{1}{3}\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Sn(1)	0·16900 (11)	0·15289 (48)	0·18447 (13)	53
Sn(2)	0·36070 (11)	0·13952 (43)	0·23063 (13)	49
Sn(3)	0·33101 (10)	0·00183 (49)	0·05599 (13)	50
Sn(4)	0·13877 (10)	0·01313 (46)	0·00923 (13)	52
C(1)	0·2699 (16)	0·3099 (49)	0·2341 (21)	57
C(2)	0·2242 (15)	0·0989 (50)	−0·0332 (20)	56
C(3)	0·1760 (21)	−0·0996 (56)	0·2748 (22)	86
C(4)	0·0858 (15)	0·3503 (69)	0·1892 (21)	91
C(5)	0·4554 (16)	0·3260 (61)	0·2785 (21)	69
C(6)	0·3850 (20)	−0·1168 (56)	0·3251 (22)	80
C(7)	0·4072 (15)	0·1223 (54)	0·0056 (19)	54
C(8)	0·3364 (19)	−0·3230 (92)	0·0525 (23)	131
C(9)	0·0389 (16)	0·1476 (60)	−0·0853 (19)	70
C(10)	0·1271 (18)	−0·3114 (73)	−0·0016 (24)	106

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

Sn(1)—Sn(4)	2·791 (3)	Sn(2)—C(6)	2·18 (4)
Sn(1)—C(1)	2·13 (3)	Sn(3)—C(2)	2·16 (3)
Sn(1)—C(3)	2·16 (4)	Sn(3)—C(7)	2·14 (3)
Sn(1)—C(4)	2·13 (4)	Sn(3)—C(8)	2·11 (6)
Sn(2)—Sn(3)	2·780 (3)	Sn(4)—C(2)	2·15 (4)
Sn(2)—C(1)	2·15 (3)	Sn(4)—C(9)	2·17 (3)
Sn(2)—C(5)	2·13 (3)	Sn(4)—C(10)	2·12 (5)
C(3)—Sn(1)—C(4)	106·2 (14)	Sn(2)—Sn(3)—C(7)	111·1 (8)
C(1)—Sn(1)—C(4)	110·2 (14)	Sn(2)—Sn(3)—C(2)	110·1 (9)
C(1)—Sn(1)—C(3)	108·0 (13)	C(7)—Sn(3)—C(8)	107·7 (15)
Sn(4)—Sn(1)—C(4)	111·4 (9)	C(2)—Sn(3)—C(8)	108·7 (14)
Sn(4)—Sn(1)—C(3)	111·3 (10)	C(2)—Sn(3)—C(7)	108·4 (12)
Sn(4)—Sn(1)—C(1)	109·6 (9)	Sn(1)—Sn(4)—C(10)	112·3 (10)
C(5)—Sn(2)—C(6)	104·8 (13)	Sn(1)—Sn(4)—C(9)	110·7 (9)
C(1)—Sn(2)—C(6)	109·7 (13)	Sn(1)—Sn(4)—C(2)	110·4 (9)
C(1)—Sn(2)—C(5)	110·2 (13)	C(9)—Sn(4)—C(10)	107·4 (14)
Sn(3)—Sn(2)—C(6)	111·1 (10)	C(2)—Sn(4)—C(10)	107·9 (14)
Sn(3)—Sn(2)—C(5)	111·0 (9)	C(2)—Sn(4)—C(9)	108·0 (12)
Sn(3)—Sn(2)—C(1)	109·9 (9)	Sn(1)—C(1)—Sn(2)	116·0 (15)
Sn(2)—Sn(3)—C(8)	110·8 (10)	Sn(3)—C(2)—Sn(4)	115·1 (14)

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Bromo(*N,N*-dimethyldithiocarbamato)dimethylgermanium and (*N,N*-Dimethyldithiocarbamato)iododimethylgermanium

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Abstract. $[Ge\{S_2CN(CH_3)_2\}Br(CH_3)_2]$, $M_r = 302{\cdot}8$, monoclinic, $P2_1$, $a = 9{\cdot}471$ (4), $b = 10{\cdot}777$ (5), $c = 11{\cdot}383$ (4) Å, $\beta = 106{\cdot}99$ (3)°, $V = 1111$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}81$, $D_m = 1{\cdot}79$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 650{\cdot}1$ mm^{-1}, $F(000) = 592$, $T = 291$ K, final $R = 0{\cdot}0519$, $wR = 0{\cdot}0543$ for 1407 unique observed reflections. $[Ge\{S_2CN(CH_3)_2\}(CH_3)_2I]$, $M_r = 349{\cdot}8$, monoclinic, $P2_1/c$, $a = 9{\cdot}935$ (3), $b = 10{\cdot}434$ (3), $c = 12{\cdot}425$ (2) Å, $\beta = 117{\cdot}71$ (2)°, $V = 1140$ (1) Å^3, $Z = 4$, $D_x = 2{\cdot}04$, $D_m = 2{\cdot}00$ Mg m^{-3}, $\mu = 543{\cdot}4$ mm^{-1}, $F(000) = 664$, $T = 291$ K, final $R = 0{\cdot}0468$, $wR = 0{\cdot}0498$ for 1235 unique observed reflections. The structure of $(CH_3)_2GeBr[S_2CN(CH_3)_2]$ contains two independent molecules in which the Ge atom is at the centre of a distorted trigonal bipyramid. Two Ge—C bonds and one Ge—S bond occupy the equatorial positions with the Ge—Br bond accounting for one of the axial positions. The other axial position is occupied by the second S atom of the dithiocarbamate ligand but at a distance much greater than in the Ge—S equatorial bond. The structure of $(CH_3)_2GeI[S_2CN(CH_3)_2]$ is similar to that just described for the bromo analogue with two notable exceptions. There is only one independent molecule and the axial Ge—S distance is considerably shorter.

Introduction. In an earlier publication (Chadha, Drake & Sarkar, 1984) we demonstrated that the structure of $(CH_3)_2GeCl[S_2CN(CH_3)_2]$ provided an example of a distorted trigonal bipyramidal germanium compound with an anisobidentate ligand. The Ge—Cl bond occupying the axial position was considerably longer and weaker than those typically found for four-coordinate germanium chlorides (Drake, Hencher & Shen, 1977; Li & Durig, 1973; Morino, Nakamaru & Iijima, 1960) and was comparable with the axial Ge—Cl bond in $[N(CH_3)_3][GeCl_4]$ (Beattie & Ozin, 1970; Bilton & Webster, 1972). A comparison of the spectroscopic data of the three compounds $(CH_3)_2GeX[S_2CN(CH_3)_2]$, X = Cl, Br, I (Chadha, Drake & Sarkar, 1986), indicated that similar distorted trigonal bipyramidal structures might be expected for the bromo and iodo derivatives. In particular, the values of the Ge—Br and Ge—I stretching vibrations were of the order of 80 and 100 cm^{-1} respectively lower than in the related four-coordinate germanium halide derivatives $(CH_3)_2GeBr_2$ and $(CH_3)_2GeI_2$ (Anderson, Barker, Drake & Hemmings, 1971; Griffiths, 1964; Van de Vondel, Van der Kelen & Van Hooydonk, 1970). We have completed the crystal structures of bromo(*N,N*-dimethyldithiocarbamato)dimethylgermanium, $(CH_3)_2GeBr[S_2CN(CH_3)_2]$, and (*N,N*-dimethyldithiocarbamato)iododimethylgermanium, $(CH_3)_2GeI[S_2CN(CH_3)_2]$, which not only confirm the predictions based on spectroscopy but also draw attention to some interesting trends.

Experimental. Preparation as described by Chadha, Drake & Sarkar (1986) followed by slow evaporation of CS_2 solution, density measured by flotation in C_2H_5I/ligroin or CH_3I/ligroin for the bromo and iodo

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