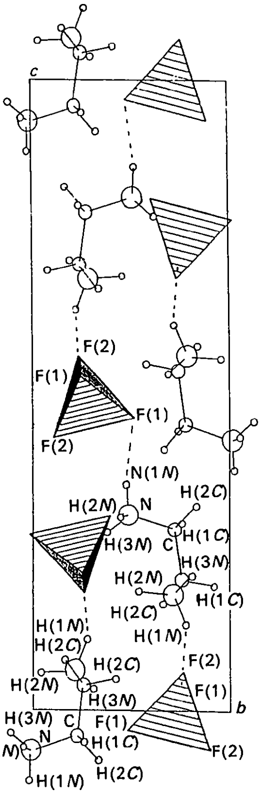

Fig. 2. Projection along the *b* axis of the atomic arrangement of $(CH_2)_2(NH_3)_2BeF_4$.

2·83 (3) Å]. The ethylenediammonium groups and BeF_4 tetrahedra alternate and form chains parallel to the [110] direction. These chains are linked together by N—H(2*N*)···F(2) bonds between an ethylenediammonium group of one chain and a tetrahedron of the other.

Fig. 2 is a projection of the atomic arrangement along the *b* axis. It shows that two (001) neighbouring planes are linked together by hydrogen bonds N—H(1*N*)···F(1) between one ethylenediammonium group in one plane to a BeF_4 tetrahedron in the other [N···F = 2·75 (3) Å]. The number and the characteristics of these bonds do not account for the existence of a cleavage parallel to (001) that was proposed by Sakurai for the sulfate, but is not observed with the fluoroberyllate crystals. The drawings were performed using the *STRUPLO*84 program (Fischer, 1985).

References

Enraf-Nonius (1977). *Structure Determination Package*. Version RSX11M. Enraf-Nonius, Delft, The Netherlands.

Fischer, R. X. (1985). *J. Appl. Cryst.* **18**, 258–262.

Main, P., Lessinger, L., Woolfson, M. M., Germain, G. & Declercq, J.-P. (1977). *MULTAN77. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

Sakurai, K. (1961). *J. Phys. Soc. Jpn*, **16**, 1205–1213.

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1,1,2,2,3,3,4,4,5,5,6,6-Dodecamethyl-1,2,4,5-tetrastannacyclohexane

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Abstract. $C_{14}H_{36}Sn_4$, $M_r = 679·20$, monoclinic, $P2_1/n$, $a = 8·236$ (3), $b = 13·584$ (5), $c = 10·738$ (3) Å, $\beta = 94·27$ (3)°, $V = 1198·0$ (7) Å^3, $Z = 2$, $D_x = 1·883$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 4·13$ mm^{-1}, $F(000) = 640$, $T = 291$ (1) K, final $R = 0·028$ for 1999 unique observed [$F \geq 3·0\sigma(F)$] diffractometer data. In the crystal the molecule resides on a centre of symmetry. The central 1,2,4,5-tetrastannacyclohexane ring has a chair conformation with an Sn—Sn bond length of 2·7753 (8) Å. The structure consists of discrete molecular units.

Introduction. Previous work (Meunier-Pieret, Van Meerssche, Gielen & Jurkschat, 1983; Preut, Bleckmann, Mitchell & Fabisch, 1984; Preut & Mitchell, 1989) indicated that while 1,2,4,5-tetrastannacyclohexanes bearing phenyl substituents at Sn prefer a chair conformation, a boat conformation is observed when the Sn atoms bear methyl substituents. In order to probe the influence of increasing substitution at the ring C atoms we have now determined the crystal structure of the title compound.

Experimental. The title compound was prepared by adding a solution of 1,3-dibromo-1,1,2,2,3,3-hexamethyl-1,3-distannapropane (0·04 mol) in ether (250 ml) to sodium (0·1 mol) in liquid ammonia

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(400 ml) at *ca* 220 K. Colourless crystals were obtained by slow recrystallization from toluene, m.p. 478 K (dec.), yield 50% (Mitchell, Fabisch, Wickenkamp, Kuivila & Karol, 1986). A well developed crystal of size ~0·46 × 0·40 × 0·42 mm was used. The crystal was mounted on a glass fibre. D_m was not determined. Intensity data were collected with $\omega/2\theta$ scans, variable scan speed 3·5–15·0° $\min^{-1}$ in θ, scan width 1·2° + dispersion. A Nicolet *R*3*m*/*V* diffractometer with graphite-monochromated Mo $K\alpha$ radiation was used. The lattice parameters were determined from symmmetry-constrained least-squares fit of 21 reflections with $2\theta_{\max} = 38{\cdot}78°$. ω scans of low-order reflections along the three crystal axes showed acceptable mosaicity. Six standard reflections (400, 060, 004, $\bar{4}00$, $0\bar{6}0$, $00\bar{4}$) were recorded every 300 reflections and an intensity loss of up to 26% was detected during 77·5 h of X-ray exposure; 4551 reflections with $2{\cdot}0 \leq 2\theta \leq 50{\cdot}0°$, $-10 \leq h \leq 10$, $0 \leq k \leq -17$, $-13 \leq l \leq 13$ were measured. The data were corrected for Lorentz–polarization, decay and absorption effects (the latter *via* ψ scans, the max./min. transmission factors were 1·00/0·83), and averaged ($R_{\text{int}} = 0{\cdot}031$) to 2127 unique reflections, 1999 of which had $F \geq 3{\cdot}0\sigma(F)$. 10 reflections were omitted because of suspected extinction. The systematic absences ($h0l$) $h + l = 2n + 1$ and ($0k0$) $k = 2n + 1$ conform to space group $P2_1/n$. The structure was solved *via* a Patterson function and $\Delta\rho$ maps. It was refined (on F) using full-matrix least squares with anisotropic displacement parameters for all non-H atoms and a common isotropic displacement parameter for the H atoms, which were placed in geometrically calculated positions (C—H 0·96 Å). 83 parameters were refined. Weights $w = 1{\cdot}0/[\sigma^2(F) + 0{\cdot}000283F^2]$ led to a featureless analysis of variance in terms of $\sin\theta$ and F_o. The refinement converged to $R = 0{\cdot}028$, $wR = 0{\cdot}031$, $S = 1{\cdot}54$ ($R = 0{\cdot}031$, $wR = 0{\cdot}043$ for all 2127 data), $(\Delta/\sigma)_{\max} = 0{\cdot}016$ (no extinction correction). The correctness of the space-group choice was checked by using *MISSYM* (Le Page, 1987). The largest peaks in the final $\Delta\rho$ map were ± 0·8 (2) e $Å^{-3}$ near Sn. Atomic scattering factors for neutral atoms and real and imaginary dispersion terms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The programs used were: *PARST* (Nardelli, 1983), *SHELXTL-Plus* (Sheldrick, 1987), *PCK*83 (Williams, 1984), *PLATON* (Spek, 1982), *MISSYM* (Le Page, 1987). The molecule and the numbering

Table 1. *Atomic coordinates and equivalent isotropic displacement parameters* ($Å^2 \times 10^4$)

$$U_{\text{eq}} = (1/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Sn(1)	0·49714 (4)	0·03276 (2)	0·20580 (3)	551
C(11)	0·7472 (9)	0·0472 (5)	0·2778 (7)	976
C(12)	0·3474 (11)	0·0638 (6)	0·3588 (6)	1111
Sn(2)	0·42686 (4)	0·16100 (2)	0·00790 (3)	471
C(21)	0·5033 (8)	0·3089 (4)	0·0577 (5)	784
C(22)	0·1704 (6)	0·1690 (5)	−0·0457 (6)	842
C(1)	0·4436 (6)	−0·1190 (3)	0·1517 (4)	527
C(2)	0·4910 (9)	−0·1880 (4)	0·2616 (5)	847
C(3)	0·2615 (7)	−0·1261 (5)	0·1160 (6)	823

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

Sn(1)—C(11)	2·155 (7)	Sn(2)—C(22)	2·150 (5)
Sn(1)—C(12)	2·168 (8)	Sn(2)—C(1*a*)	2·162 (5)
Sn(1)—Sn(2)	2·7753 (8)	C(1)—C(2)	1·535 (7)
Sn(1)—C(1)	2·177 (5)	C(1)—C(3)	1·524 (7)
Sn(2)—C(21)	2·161 (6)		
Sn(2)—Sn(1)—C(1)	111·3 (1)	C(21)—Sn(2)—C(1*a*)	106·8 (2)
C(12)—Sn(1)—C(1)	105·7 (2)	Sn(1)—Sn(2)—C(1*a*)	110·7 (2)
C(12)—Sn(1)—Sn(2)	111·1 (2)	C(22)—Sn(2)—C(1*a*)	109·5 (2)
C(11)—Sn(1)—C(1)	110·6 (2)	Sn(1)—C(1)—C(3)	107·7 (3)
C(11)—Sn(1)—Sn(2)	110·9 (2)	Sn(1)—C(1)—C(2)	109·8 (3)
C(11)—Sn(1)—C(12)	107·0 (3)	C(2)—C(1)—C(3)	110·1 (5)
Sn(1)—Sn(2)—C(22)	112·4 (2)	Sn(1)—C(1)—Sn(2*a*)	111·0 (2)
Sn(1)—Sn(2)—C(21)	110·7 (2)	C(2)—C(1)—Sn(2*a*)	109·6 (3)
C(21)—Sn(2)—C(22)	106·3 (2)	C(3)—C(1)—Sn(2*a*)	108·6 (3)
Sn(2)—Sn(1)—C(1)—Sn(2*a*)	56·1 (2)	Sn(2*a*)—Sn(1*a*)—C(1*a*)—Sn(2)	−56·1 (2)
Sn(1)—C(1)—Sn(2*a*)—Sn(1*a*)	−55·8 (2)	Sn(1*a*)—C(1*a*)—Sn(2)—Sn(1)	55·8 (2)
C(1)—Sn(2*a*)—Sn(1*a*)—C(1*a*)	55·9 (2)	C(1*a*)—Sn(2)—Sn(1)—C(1)	−55·9 (2)

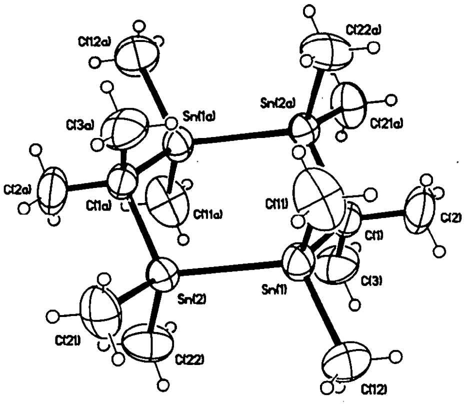

Fig. 1. General view (*SHELXTL-Plus*) of the molecule, showing the atom-numbering scheme. Anisotropic ellipsoids represent 50% probability boundaries.

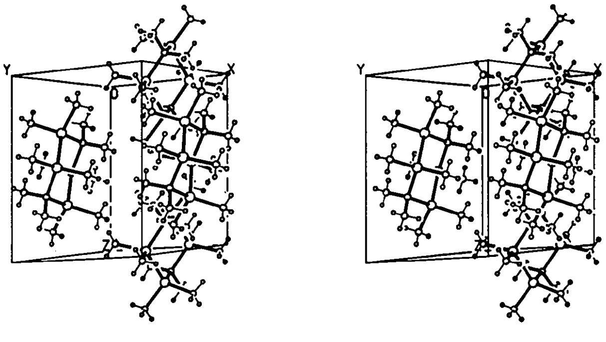

Fig. 2. Stereoscopic view (*SHELXTL-Plus*) of the unit cell plus two molecules.

scheme are shown in Fig. 1 and a stereoscopic view of the unit cell and four molecules is given in Fig. 2. Positional parameters and the equivalent isotropic values of the anisotropic displacement parameters for the non-H atoms are given in Table 1.* Bond lengths, bond angles and torsion angles are given in Table 2.

Discussion. The important feature of the structure revealed by the present study is the transition of the 1,2,4,5-tetrastannacyclohexane ring from the boat forms observed previously (Preut, Bleckmann, Mitchell & Fabisch, 1984; Preut & Mitchell, 1989) to a chair form. The presence of two methyl groups on each ring C atom apparently destabilizes the boat form because of unfavourable 1,4-axial methyl interactions similar to the 1,4-hydrogen interaction which destabilizes the boat form of cyclohexane. While the C—Sn—C angles vary only slightly between the octa-, deca- and dodecamethyl tetrastannacyclohexanes, the Sn—C—Sn angle decreases steadily [average values: octamethyl 115·5 (15), decamethyl 112·2 (4)°]. Although the torsion angles are similar for the two boat forms [octamethyl: 0 (1), 5 (2), −54 (2), 0 (1), 55 (2), −55 (2)°; *cis*-decamethyl: −1·5 (1), 51·7 (1), −48·8 (1), −3·5 (1), 54·6 (1), −51·5 (1)°] they are very different for the two chair structures [octaphenyl: −39·2 (3), 42·7 (4), −42·3 (4), 39·2 (3), −42·7 (4), 42·3 (4)°; title compound: −55·9 (2), 55·8 (2), −56·1 (2), 55·9 (2), −55·8 (2), 56·1 (2)°].

References

Le Page, Y. (1987). *J. Appl. Cryst.* **20**, 264–269.

Meunier-Piret, J., Van Meerssche, M., Gielen, M. & Jurkschat, K. (1983). *J. Organomet. Chem.* **252**, 289–294.

Mitchell, T. N., Fabisch, B., Wickenkamp, R., Kuivila, H. G. & Karol, T. (1986). *Silicon Germanium Tin Lead Compd.* **9**, 57–66.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.

Preut, H., Bleckmann, P., Mitchell, T. & Fabisch, B. (1984). *Acta Cryst.* **C40**, 370–372.

Preut, H. & Mitchell, T. (1989). *Acta Cryst.* **C45**, 35–37.

Sheldrick, G. M. (1987). *SHELXTL-Plus.* Release 3.4 for Nicolet *R3m/V* crystallographic systems. *An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* Univ. of Göttingen, Germany.

Spek, A. L. (1982). *The EUCLID Package.* In *Computational Crystallography*, edited by D. Sayre, p. 528. Oxford: Clarendon Press.

Williams, D. E. (1984). *PCK83. A Crystal and Molecular Packing Analysis Program.* QCPE Program No. 481. Department of Chemistry, Indiana Univ., USA.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53716 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Bis(*N*,*N*-dimethyl-1,2-ethanediamine)platinum(II) Diiodide

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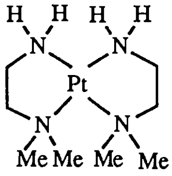
Abstract. $[Pt(C_8H_{24}N_4)]I_2$, $M_r = 625·2$, monoclinic, $P2_1/c$, $a = 5·712\ (1)$, $b = 15·901\ (5)$, $c = 8·740\ (1)$ Å, $\beta = 93·263\ (9)°$, $V = 792·5$ Å^3, $Z = 2$, $D_m = 2·62\ (2)$, $D_x = 2·62$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 127·9$ cm^{-1}, $F(000) = 568$, $T = 293$ K, final $R = 0·037$ for 1882 counter-measured reflections. The structure consists of centrosymmetric square-planar molecules. Pt—N distances are 2·053 (5) and 2·045 (5) Å and the chelate N—Pt—N angle is 83·7 (2)°.

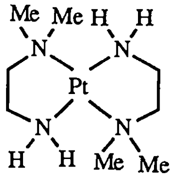
Introduction. The complex bis(*N*,*N*-dimethyl-1,2-ethanediamine)platinum(II) diiodide, $[Pt(dmen)_2]I_2$, has been prepared by reaction (1).

$$Pt(dmen)I_2 + dmen \rightarrow [Pt(dmen)_2]I_2. \qquad (1)$$

cis (*A*) or *trans* (*B*) stereochemistry is possible for the complex cation, and this cannot be readily determined by spectroscopic and physicochemical methods. Thus, the X-ray crystal structure of the compound has been determined and the X-ray powder pattern of the bulk material has been compared with the single-crystal measurements to ascertain whether the synthesis is stereoselective.



(*A*)



(*B*)

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