

Fig. 4. Crystal structure viewed down the  $c$  axis. The molecular columns I, II and III are packed with van der Waals contacts.

The  $c$ -axis projection of the crystal structure is shown in Fig. 4, where the molecular columns I, II and III are related by translations; they are displaced by  $\pm c/3$  along the columns and fill in the space with each other. Shortest intermolecular distances are summarized in Table 3.

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## 2,2-Dimethyl-1,3-dithia-2-stannacyclopentane, $C_4H_{10}S_2Sn$

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**Abstract.**  $M_r = 240.93$ , orthorhombic,  $P2_12_12_1$ ,  $a = 6.6906$  (2),  $b = 9.2514$  (3),  $c = 12.5897$  (4) Å,  $V = 779.27$  (5) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 2.053$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 34.5$  cm<sup>-1</sup>,  $F(000) = 464$ ,  $T = 294$  K. Final  $R = 0.022$  for 1982 observed reflections. The structure contains molecules with a five-membered  $\text{SnS}_2\text{C}_2$  ring (envelope conformation), with a short intermolecular  $\text{Sn}\cdots\text{S}$  contact completing distorted trigonal bipyramidal coordination at tin.  $\text{Sn}-\text{S} = 2.415$  (1), 2.474 (1),  $\text{Sn}\cdots\text{S} = 3.182$  (1),  $\text{Sn}-\text{CH}_3 = 2.130$  (3),  $\text{S}-\text{C} = 1.832$  (4),  $\text{CH}_2-\text{CH}_2 = 1.509$  (7) Å.

**Introduction.** 2,2-Dimethyl-1,3-dithia-2-stannacyclopentane,  $(\text{CH}_3)_2\text{SnS}(\text{CH}_2)_2\text{S}$ , is obtained by reaction of dichlorodimethyltin and 1,2-ethanedithiol (Wieber & Schimdt, 1963; Abel & Brady, 1965; Bernardi, Di-stefano, Modelli, Pietropaolo & Ricci, 1977). The present paper describes the crystal structure.

**Experimental.** Crystal dimensions  $0.2 \times 0.2 \times 0.4$  mm, CAD-4F diffractometer, Mo  $K\alpha$  radiation (graphite

The calculations were performed on a FACOM M200 computer of this Institute using the UNICS III program system (Sakurai & Kobayashi, 1979). The authors are grateful to Professor T. Shimozawa, Saitama University, for his encouragement throughout this work.

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monochromator),  $hkl$ ,  $0 < \theta \leq 40.0^\circ$ ,  $\omega - (4/3)\theta$  scans,  $\omega$  scan angle  $(0.60 + 0.35 \tan \theta)^\circ$  (extended by 25% on each side for background counts), scan speeds  $1.18$ – $10.06^\circ \text{ min}^{-1}$ , horizontal aperture  $(2.00 + \tan \theta)$  mm, vertical aperture 4 mm; intensity check reflections every hour (decrease of 1% over the whole period) and crystal orientation checked after every 100 reflections; cell parameters by least-squares fit to the  $\sin \theta$  values for 24 reflections with  $40 \leq 2\theta \leq 52^\circ$ ; intensities corrected for Lorentz and polarization effects and for absorption (Coppens, Leiserowitz & Rabinovich, 1965; Busing & Levy, 1967), transmission factors 0.456–0.590; of 2722 reflections measured, 1982 (73%) had  $I \geq 3\sigma(I)$ , where  $\sigma^2(I) = S + 2B + [0.04(S-B)]^2$ ,  $S$  = scan count,  $B$  = time-averaged background count. Structure determined by Patterson and Fourier methods; refined by full-matrix least-squares techniques, finally with anisotropic thermal parameters; H atoms located on a difference map and included in refinement with isotropic temperature factors; function minimized  $\sum w(|F_o| - |F_c|)^2$ , with

$w = 1/\sigma^2(F)$ , where  $\sigma^2(F)$  is derived from the previously defined  $\sigma^2(I)$ , for unobserved reflections  $w = 0$ ; isotropic type I extinction correction applied (Becker & Coppens, 1974, 1975; Coppens & Hamilton, 1970; Thornley & Nelmes, 1974), final value of  $g = 1.22(8) \times 10^4$ ; convergence reached at  $R = 0.022$ ,  $R_w = 0.029$  for the 1982 observed reflections (the opposite enantiomorph refined to  $R = 0.026$ ,  $R_w = 0.034$ , so that the present results correspond to the correct absolute configuration for the particular crystal examined); for all 2722 reflections,  $R = 0.041$ ; on final cycle of refinement mean and maximum parameter shifts were  $0.1\sigma$  and  $1.0\sigma$ ; mean error in an observation of unit weight was  $0.97$ ; final difference map had maximum fluctuations of  $+1.7$  and  $-1.0 e \text{ \AA}^{-3}$ . Final positional parameters are given in Table 1.\*

Analysis of the thermal motion (Schomaker & Trueblood, 1969) indicates that the five-membered ring behaves as a rigid body, with some additional riding motion of the methyl groups; bond lengths (Table 2) were corrected accordingly (Cruickshank, 1956, 1961).

Atomic scattering factors from Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965), with anomalous-dispersion corrections for Sn and S (Cromer & Liberman, 1970); computer programs used include locally written programs for data processing and locally modified versions of the following: *ORFLS* (Busing, Martin & Levy, 1962); *ORFFE* (Busing, Martin & Levy, 1964); *FORDAP* (A. Zalkin); and *ORTEP II* (Johnson, 1976).

**Discussion.** The crystal contains molecules with a five-membered  $\text{SnS}_2\text{C}_2$  ring and a coordination at the  $\text{Sn}^{\text{IV}}$  atom which could be described as distorted tetrahedral [angles  $89.55(3)$ – $121.7(2)^\circ$ ]. However there is a short intermolecular  $\text{Sn}\cdots\text{S}(2')$  contact which completes five-coordination at Sn, the geometry being best described as a distorted trigonal bipyramid with S(1), C(3), C(4) equatorial and S(2), S(2') axial (Fig. 1). The  $\text{S}(2)\text{—Sn}\cdots\text{S}(2')$  angle is  $162.33(3)^\circ$ , the equatorial angles are  $\text{S}(1)\text{—Sn—C}(3)$ ,  $\text{S}(1)\text{—Sn—C}(4)$ ,  $\text{C}(3)\text{—Sn—C}(4) = 111.3(1)$ ,  $120.0(1)$ ,  $121.7(2)^\circ$ , respectively, and the  $\text{ax—Sn—eq}$  angles are  $89.55(3)$ ,  $102.4(1)$ ,  $103.9(1)^\circ$  involving S(2), and  $74.27(3)$ ,  $80.4(1)$ ,  $88.9(1)^\circ$  involving S(2') (Table 2).

The Sn—S bonds in the molecule differ significantly, Sn—S(1) (equatorial) =  $2.415(1)$  and Sn—S(2) (axial) =  $2.474(1)$  Å ( $2.429$  and  $2.485$  Å after libration correction), in comparison with a covalent-radii sum of  $2.44$  Å (Pauling, 1960). The intermolecular

$\text{Sn}\cdots\text{S}(2')$  [ $-x, -\frac{1}{2} + y, -\frac{1}{2} - z$ ] contact is  $3.182(1)$  Å, much shorter than the normal van der Waals contact of about  $4.0$  Å (Bondi, 1964).

Table 1. Final positional (fractional,  $\times 10^5$  for Sn, S;  $\times 10^4$  for C;  $\times 10^3$  for H) and isotropic thermal parameters ( $U \times 10^3 \text{ \AA}^2$ ) with e.s.d.'s in parentheses

	x	y	z	$U_{\text{eq}}^*/U_{\text{iso}}$
Sn	-6368 (3)	-9197 (2)	-22543 (1)	32
S(1)	29609 (13)	-11351 (10)	-22621 (9)	47
S(2)	-3368 (15)	13818 (9)	-12670 (6)	39
C(1)	3599 (6)	421 (5)	-1432 (4)	52
C(2)	2029 (8)	866 (5)	-636 (3)	52
C(3)	-1954 (7)	-2364 (4)	-1132 (3)	44
C(4)	-2167 (7)	-275 (5)	-3655 (3)	44
H1(1)	389 (6)	126 (5)	-193 (3)	28 (9)
H2(1)	471 (7)	20 (5)	-102 (4)	47 (12)
H1(2)	169 (7)	10 (5)	-19 (4)	52 (12)
H2(2)	241 (15)	166 (9)	-28 (7)	133 (34)
H1(3)	-352 (12)	-259 (9)	-130 (6)	100 (21)
H2(3)	-232 (18)	-228 (12)	-60 (8)	137 (41)
H3(3)	-137 (21)	-306 (14)	-119 (11)	192 (62)
H1(4)	-260 (10)	63 (6)	-355 (5)	68 (17)
H2(4)	-323 (9)	-72 (6)	-370 (4)	50 (13)
H3(4)	-129 (11)	-52 (7)	-433 (6)	84 (22)

\*  $U_{\text{eq}}$  = one third the trace of the diagonalized anisotropic temperature factor matrix.

Table 2. Bond lengths (Å) and angles ( $^\circ$ ) with e.s.d.'s in parentheses

	Uncorrected	Corrected		Uncorrected	Corrected
Sn—S(1)	2.415 (1)	2.429	S(1)—C(1)	1.830 (5)	1.838
Sn—S(2)	2.474 (1)	2.485	S(2)—C(2)	1.834 (5)	1.845
Sn—C(3)	2.135 (4)	2.145	C(1)—C(2)	1.509 (7)	1.519
Sn—C(4)	2.125 (4)	2.132	Sn $\cdots$ S(2')	3.182 (1)	—
S(1)—Sn—S(2)	89.55 (3)		C(3)—Sn $\cdots$ S(2')	88.89 (13)	
S(1)—Sn—C(3)	111.26 (13)		C(4)—Sn $\cdots$ S(2')	80.39 (13)	
S(1)—Sn—C(4)	120.00 (13)		C(3)—Sn—C(4)	121.73 (18)	
S(2)—Sn—C(3)	103.87 (11)		Sn—S(1)—C(1)	99.52 (14)	
S(2)—Sn—C(4)	102.39 (11)		Sn—S(2)—C(2)	93.65 (13)	
S(2)—Sn $\cdots$ S(2')	162.33 (3)		S(1)—C(1)—C(2)	115.57 (31)	
S(1)—Sn $\cdots$ S(2')	74.27 (3)		S(2)—C(2)—C(1)	112.61 (26)	

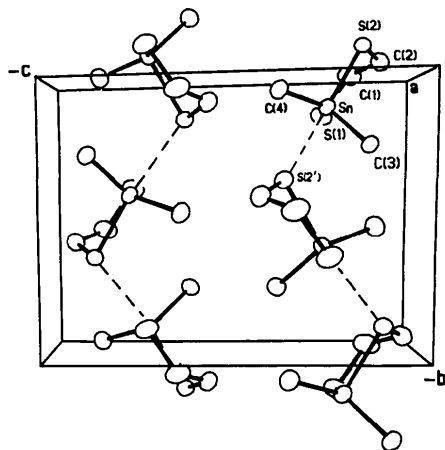


Fig. 1. Structure of 2,2-dimethyl-1,3-dithia-2-stannacyclopentane.

\* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles involving H atoms, torsion angles, and a molecular diagram have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38301 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

These dimensions and configuration are similar to those observed in a variety of Sn—S compounds. Dimethyldithiocarbamate derivatives of Sn<sup>IV</sup> exhibit five- and six-coordination for Sn, with bonding which varies from symmetric bidentate to unidentate, often with additional weaker Sn...S interactions. *E.g.* in the orthorhombic and monoclinic forms of Me<sub>3</sub>SnS<sub>2</sub>CNMe<sub>2</sub> (Sheldrick & Sheldrick, 1970; Sheldrick, Sheldrick, Dalton & Jones, 1970), Sn atoms have trigonal bipyramidal coordinations with two Me and one S equatorial [Sn—S = 2.47 (1) Å] and one Me and one S axial [Sn...S = 3.16–3.33 (1) Å]. In Me<sub>2</sub>ClSnS<sub>2</sub>CNMe<sub>2</sub> (Furue, Kimura, Yasuoka, Kasai & Kakudo, 1970) the Sn—S distances are more symmetrical at 2.48 (1) (equatorial), 2.79 (1) Å (axial). 2,2-Dichloro-1,3,6,2-trithiastannocane (Dräger & Engler, 1975) has two equatorial Sn—S = 2.387 (3) Å, with a transannular S occupying the axial position of a trigonal bipyramid, Sn...S = 2.760 (3) Å. Pseudo-six-coordination for tin is found in Me<sub>2</sub>Sn[S<sub>2</sub>CNMe<sub>2</sub>]<sub>2</sub> (Kimura, Yasuoka, Kasai & Kakudo, 1972) and Ph<sub>2</sub>Sn[S<sub>2</sub>P(OEt)<sub>2</sub>]<sub>2</sub> (Liebich & Tomassini, 1978), with Sn—S = 2.48–2.52 (1), Sn...S = 2.95–3.23 (1) Å.

The difference between the Sn—S (equatorial) and Sn—S (axial) bonds in the present compound, 2.415 (1) and 2.474 (1) Å, respectively, is typical for trigonal bipyramidal coordination for a non-transition metal. The Sn—CH<sub>3</sub> distances, 2.125 (4) and 2.135 (4) Å (2.132 and 2.145 Å after libration correction) do not differ significantly, mean 2.130 (3) Å (2.139 Å), and are similar to distances observed in other methyltin compounds; the covalent-radius sum is 2.17 Å (Pauling, 1960). The S—C bonds are also normal, mean = 1.832 (5) Å (1.842 Å after correction), sum of covalent radii = 1.81 Å. The CH<sub>2</sub>—CH<sub>2</sub> bond, 1.509 (7) Å (1.519 Å after correction), is not significantly shorter than a normal single bond.

The conformation of the five-membered ring is best described as an envelope with C(2) displaced by about 0.9 Å from the approximate plane of the other four atoms [the equation of the Sn, S(1), S(2) plane is 0.2539x + 4.6359y - 10.885z = 2.0111, with C(2) displaced 0.866 (4) Å, and C(1) displaced in the same direction by 0.166 (5) Å]; the ring torsion angles\* are: Sn—S(1) = 5.3 (1), Sn—S(2) = -28.2 (1), S(1)—C(1) = 27.3 (3), S(2)—C(2) = 54.4 (3), and C(1)—C(2) = -60.5 (3)°. The ring conformation is thus very asymmetric relative to the C<sub>2</sub> or C<sub>2v</sub> symmetries which might be expected for the free molecule, the torsion angles being more nearly sym-

metric about a line from C(2) to the midpoint of Sn—S(1).

The short Sn...S(2') interaction links molecules into chains along **b** (Fig. 1) with normal van der Waals contacts between chains.

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\* See previous footnote.