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Two Crystal Modifications of Hexamethylcyclotristannatellurane

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Abstract. $[(CH_3)_2SnTe]_3$, $C_6H_{18}Sn_3Te_3$, M_r = 829·1, Mo $K\alpha$ radiation, λ = 0·71069 Å, T = 293 K: (1) monoclinic, $P2_1/c$, a = 14·681 (2), b = 10·2775 (8), c = 13·561 (3) Å, β = 112·087 (12)°, V = 1896·0 Å^3, Z = 4, D_x = 2·905 Mg m^{-3}, μ = 8·45 mm^{-1}, $F(000)$ = 1439·9, R = 0·034 for 1968 observed data; (2) tetragonal, $I4_1/a$, a = 12·710 (3), c = 22·757 (4) Å, V = 3676·3 Å^3, Z = 8, D_x = 2·996 Mg m^{-3}, μ = 8·72 mm^{-1}, $F(000)$ = 2879·8, R = 0·024 for 1507 observed data. In each form the molecule adopts a twist-boat conformation which, in the case of the tetragonal modification, displays crystallographic twofold symmetry.

Experimental. $[(CH_3)_2SnTe]_3$ was obtained in petroleum solution by Soxhlet extraction of the crude reaction product (Blecher & Dräger, 1979; Gay, Jones & Sharma, 1989). Evaporation of the solvent at room temperature yielded a sample consisting mainly of thin yellow-green sheets and a few thicker plates of amber colour. The solid state ^{119}Sn and ^{125}Te NMR spectra (Gay *et al.*, 1989) displayed multiple resonances suggesting that more than one crystalline form was present. A plate selected from this sample for X-ray analysis was found to have crystallized in the space group $P2_1/c$. A different yellow crystal modification was formed exclusively when a saturated petroleum solution of $[(CH_3)_2SnTe]_3$ was cooled to 255 K, as confirmed by its solid state ^{119}Sn and ^{125}Te NMR spectra (Gay *et al.*, 1989). X-ray analysis showed these crystals to be tetragonal, space group $I4_1/a$. Attempts to convert this phase to the monoclinic form by heating were unsuccessful. At 343 K the solid material appeared to fuse to give an apparently polymeric material of negligible solubility in petroleum ether. In either case the crystals became black upon exposure to air for several mintues.

Crystals were mounted on Pyrex filaments with five-minute epoxy resin and were protected from air exposure by a thin coating of resin. Cell-parameter determination (25 reflections 15·1 ≤ θ ≤ 19·1°, $P2_1/c$; 24 reflections 20·3 ≤ θ ≤ 25·7°, $I4_1/a$) and data acquisition were performed using an Enraf–Nonius CAD-4F diffractometer with Mo $K\alpha$ radiation and a graphite monochromator. The symmetry of the intensity-weighted reciprocal lattices and the systematic absences were determined using the diffractometer. Two standard intensities were measured every hour of acquisition time and showed no systematic variations.

An empirical absorption correction (North, Phillips & Mathews, 1968) was applied to the data in the case of the $I4_1/a$ modification because the irregular shape of the crystal made measurement of crystal faces impracticable. The correction factors applied to the intensities ranged from 1·0 to 0·813. In the $P2_1/c$ case, an analytical absorption correction (de Meulenaer & Tompa, 1965) was made (transmission coefficients ranging from 0·614 to 0·248). Data reduction, including Lorentz and polarization corrections, was performed.

In both cases the coordinates for the Te and Sn atoms were determined by direct methods (Main, Woolfson & Germain, 1971). The C-atom positions were revealed by electron density difference Fourier syntheses. H atoms were fixed at calculated positions with assigned isotropic temperature factors, basing the rotational conformation of each CH_3 group upon the positions of the strongest peaks near the C atoms from a difference Fourier map. The final, full-matrix least-squares refinement, minimizing

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Table 1. *Data-acquisition and refinement details for two modifications of* $[(\mathrm{CH_3})_2\mathrm{SnTe}]_3$

	$P2_1/c$	$I4_1/a$
Crystal dimensions (mm)	0·06 × 0·22 × 0·27	0·33 × 0·35 × 0·36
Scan mode	ω–2θ	ω–2θ
Scan width (°)	0·8 + 0·345 tan θ	0·9 + 0·345 tan θ
Scan speeds (° $\mathrm{min^{-1}}$)	0·75–3·3	0·79–3·3
Range 2θ (°)	3–55	3–55
Reflections (unique measured)	4328 ($\pm h, k, l$: $l = 0$ $h > 0$)	2109 (h, k, l: $h + k + l = 2n$)
Reflections [$I \geq 2{\cdot}5\sigma(I)$]	1968	1507
Refined parameters	109	57
R	0·034	0·024
wR $\{w = [\sigma^2(F) + 0{\cdot}004F^2]^{-1}\}$	0·040	0·034
S	1·22	1·20

Table 2. *Fractional atomic coordinates and equivalent isotropic thermal parameters for* $[(\mathrm{CH_3})_2\mathrm{SnTe}]_3$, $P2_1/c$

$$B_{\mathrm{eq}} = (8\pi^2/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq} (Å^2)
Te(1)	0·42376 (7)	0·22412 (9)	0·37728 (7)	5·40
Te(2)	0·35208 (6)	−0·19034 (9)	0·31460 (7)	4·40
Te(3)	0·14041 (8)	0·11416 (10)	0·09903 (7)	5·40
Sn(1)	0·18030 (6)	−0·14277 (9)	0·14724 (6)	4·24
Sn(2)	0·22357 (7)	0·21227 (9)	0·30073 (7)	4·70
Sn(3)	0·45760 (6)	0·00962 (9)	0·27616 (6)	4·05
C(11)	0·0686 (10)	−0·2243 (17)	0·1928 (12)	6·9
C(12)	0·1801 (13)	−0·2302 (17)	0·0028 (11)	7·9
C(21)	0·1727 (10)	0·0942 (13)	0·3984 (10)	5·4
C(22)	0·1782 (14)	0·4121 (16)	0·2909 (15)	8·9
C(31)	0·4158 (10)	0·0432 (13)	0·1088 (10)	5·3
C(32)	0·6108 (10)	−0·0257 (14)	0·3642 (10)	5·4

Table 3. *Fractional atomic coordinates and equivalent isotropic thermal parameters for* $[(\mathrm{CH_3})_2\mathrm{SnTe}]_3$, $I4_1/a$

$$B_{\mathrm{eq}} = (8\pi^2/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq} (Å^2)
Te(1)	0·00000	0·25000	0·49811 (3)	4·03
Te(2)	−0·17585 (4)	0·22318 (4)	0·32838 (2)	4·07
Sn(1)	0·00000	0·25000	0·26103 (3)	3·70
Sn(2)	−0·08343 (4)	0·11278 (4)	0·41774 (2)	3·34
C(1)	0·0267 (8)	0·1153 (9)	0·2077 (4)	6·6
C(2)	−0·2037 (7)	0·0271 (6)	0·4630 (4)	4·9
C(3)	0·0327 (7)	0·0061 (7)	0·3855 (4)	5·3

Table 4. *Selected distances* (Å) *and angles* (°) *for* $[(\mathrm{CH_3})_2\mathrm{SnTe}]_3$, $P2_1/c$

Te(1)—Sn(2)	2·7283 (15)	Sn(1)—C(11)	2·130 (16)
Te(1)—Sn(3)	2·7380 (14)	Sn(1)—C(12)	2·154 (14)
Te(3)—Sn(1)	2·7305 (14)	Sn(2)—C(22)	2·147 (16)
Te(2)—Sn(1)	2·7287 (13)	Sn(3)—C(32)	2·144 (13)
Te(3)—Sn(2)	2·7345 (14)	Sn(2)—C(21)	2·128 (15)
Te(2)—Sn(3)	2·7390 (13)	Sn(3)—C(31)	2·144 (12)
Sn(2)—Te(1)—Sn(3)	98·37 (4)	C(22)—Sn(2)—Te(1)	104·2 (5)
Sn(1)—Te(3)—Sn(2)	98·17 (4)	C(32)—Sn(3)—Te(1)	102·0 (4)
Sn(1)—Te(2)—Sn(3)	96·85 (4)	C(22)—Sn(2)—Te(3)	106·2 (5)
Te(3)—Sn(1)—Te(2)	114·86 (4)	C(32)—Sn(3)—Te(2)	108·5 (4)
Te(1)—Sn(2)—Te(3)	114·14 (5)	C(21)—Sn(2)—Te(1)	111·6 (4)
Te(1)—Sn(3)—Te(2)	106·68 (4)	C(31)—Sn(3)—Te(1)	111·5 (4)
C(11)—Sn(1)—Te(3)	109·3 (5)	C(21)—Sn(2)—Te(3)	106·5 (4)
C(12)—Sn(1)—Te(2)	111·5 (5)	C(31)—Sn(3)—Te(2)	111·4 (4)
C(11)—Sn(1)—Te(2)	105·1 (4)	C(11)—Sn(1)—C(12)	111·2 (8)
C(12)—Sn(1)—Te(3)	105·0 (5)	C(22)—Sn(2)—C(21)	114·3 (6)
		C(32)—Sn(3)—C(31)	116·1 (5)

Torsion angles (°)

Sn(3)—Te(1)—Sn(2)—Te(3)	−32·59 (6)	Te(1)—Sn(3)—Te(2)—Sn(1)	87·19 (5)
Sn(2)—Te(1)—Sn(3)—Te(2)	−50·04 (5)	Te(2)—Sn(1)—Te(3)—Sn(2)	−33·26 (6)
Te(1)—Sn(2)—Te(3)—Sn(1)	76·58 (6)	Te(3)—Sn(1)—Te(2)—Sn(3)	−35·26 (5)

Table 5. *Selected distances* (Å) *and angles* (°) *for* $[(\mathrm{CH_3})_2\mathrm{SnTe}]_3$, $I4_1/a$

Primed atoms are related to the unprimed by $-x, \frac{1}{2}-y, z$.

Te(1)—Sn(2)	2·7407 (8)	Sn(1)—C(1)	2·126 (9)
Te(2)—Sn(1)	2·7314 (8)	Sn(2)—C(2)	2·140 (8)
Te(2)—Sn(2)	2·7358 (8)	Sn(2)—C(3)	2·134 (8)
Sn(2)—Te(1)—Sn(2′)	96·28 (3)	Te(2)—Sn(1)—Te(2′)	111·73 (3)
Sn(1)—Te(2)—Sn(2)	97·46 (3)	Te(1)—Sn(2)—Te(2)	109·62 (3)
C(1)—Sn(1)—Te(2)	110·5 (3)	C(3)—Sn(2)—Te(1)	111·5 (3)
C(1)—Sn(1)—Te(2′)	106·9 (3)	C(3)—Sn(2)—Te(2)	111·5 (3)
C(2)—Sn(2)—Te(1)	106·2 (3)	C(1)—Sn(1)—C(1′)	110·4 (4)
C(2)—Sn(2)—Te(2)	108·2 (3)	C(2)—Sn(2)—C(3)	109·7 (3)

Torsion angles (°)

Te(2)—Sn(2)—Te(1)—Sn(2′)	−42·25 (3)	Te(2′)—Sn(1)—Te(2)—Sn(2)	−35·22 (3)
Sn(1)—Te(2)—Sn(2)—Te(1)	86·00 (3)		

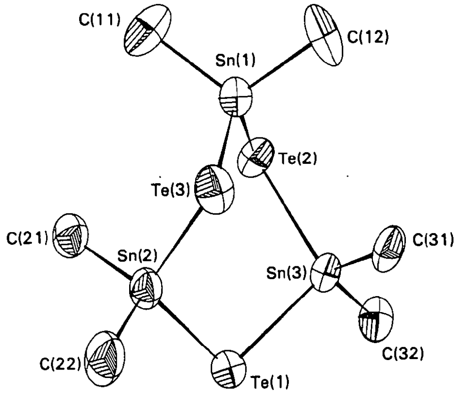

Fig. 1. Molecular structure of $[(\mathrm{CH_3})_2\mathrm{SnTe}]_3$, $P2_1/c$; 50% enclosure thermal ellipsoids; H atoms not shown.

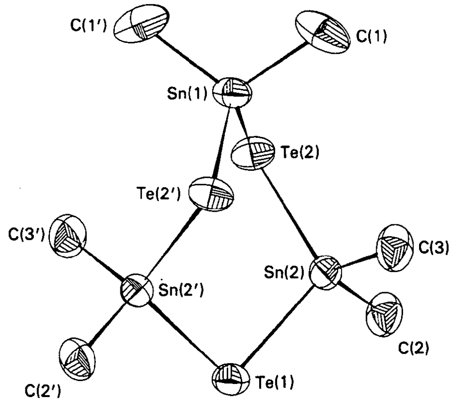

Fig. 2. Molecular structure of $[(\mathrm{CH_3})_2\mathrm{SnTe}]_3$, $I4_1/a$; 50% enclosure thermal ellipsoids; H atoms not shown.

$\sum w(|F_o| - |F_c|)^2$, in each case included justified anisotropic thermal parameters for the non-H atoms. In the $I4_1/a$ case a refined extinction variable [$g = 2{\cdot}8\ (2) \times 10^{-7}$] (Larson, 1970) was included resulting in a marked improvement in the residual. The refinements were terminated when the maximum shift/e.s.d. dropped below 0·01. The final difference map for the $P2_1/c$ structure had a largest peak of 0·8 (2) e Å^{-3}, 1·13 Å from Te(1). For the $I4_1/a$ case, the largest peak was 0·9 (2) e Å^{-3}, 0·89 Å fromTe(2). A summary of details for data acquisition and structure refinement is given in Table 1.

The programs used for data reduction and structure refinement were from the *NRCVAX* crystal structure system (Gabe, Larson, Lee & Le Page, 1984) and used scattering factors and anomalous-dispersion corrections for neutral atoms (*International Tables for X-ray Crystallography,* 1974). Computations were performed with a MicroVAXII computer. Diagrams were generated by the program *SNOOPI* (Davis, 1985). The final positional and equivalent isotropic thermal parameters for the non-H atoms are given in Table 2 ($P2_1/c$) and Table 3 ($I4_1/a$). Selected bond distances and angles are given in Table 4 ($P2_1/c$) and Table 5 ($I4_1/a$).* The molecular structures are shown in Fig. 1 ($P2_1/c$) and Fig. 2 ($I4_1/a$).

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* Tables of anisotropic thermal parameters, calculated H-atom coordinates and measured and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52073 (29 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Nitratobis(triphenylphosphine)silver(I) Benzene Solvate (1/1)

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Abstract. $[Ag(C_{18}H_{15}P)_2NO_3].C_6H_6$, $M_r = 772{\cdot}5$, triclinic, $P\bar{1}$, $a = 12{\cdot}279\ (1)$, $b = 16{\cdot}260\ (1)$, $c = 11{\cdot}134\ (1)$ Å, $\alpha = 102{\cdot}51\ (1)$, $\beta = 114{\cdot}28\ (1)$, $\gamma = 101{\cdot}79\ (1)^\circ$, $U = 1868\ (1)$ Å^3, $Z = 2$, $D_x = 1{\cdot}373$ Mg m^{-3}, Mo $K\bar{\alpha}$ radiation, $\lambda = 0{\cdot}7107$ Å, $\mu = 0{\cdot}623$ mm^{-1}, $F(000) = 792$, $T = 295\ (2)$ K, $R = 0{\cdot}025$ for 5139 observed reflections. The Ag atom in the title compound is coordinated by two P atoms [Ag—P(1) 2·435 (1), Ag—P(2) 2·416 (1) Å, P(1)—Ag—P(2) 139·4 (1)°] and two O atoms derived from a weakly associated nitrate group [Ag—O(1) 2·463 (2), Ag—O(2) 2·572 (2) Å]. In the crystal lattice, layers of solvent benzene molecules are sandwiched between layers of complex molecules.

Experimental. The title compound was the major isolable product from the reaction of [Ag(adeninate)]NO_3 and triphenylphosphine (1/2) in refluxing $CHCl_3$ solution (Lobana, Bhatia & Tiekink, 1989). Crystals were obtained from benzene solution as a (1/1) benzene solvate; m.p. 394–395 K. Enraf–Nonius CAD-4F diffractometer controlled by a PDP8/A computer, graphite-monochromated Mo $K\bar{\alpha}$ radiation; ω:2θ scan technique. Cell parameters on crystal $0{\cdot}44 \times 0{\cdot}31 \times 0{\cdot}65$ mm by least squares on 25 reflections ($8 \leq \theta \leq 12^\circ$) (de Boer & Duisenberg, 1984). Analytical absorption correction (Sheldrick, 1976) applied; max. and min. transmission factors 0·838 and 0·727. Total of 7524 reflections ($1{\cdot}5 \leq \theta \leq 25{\cdot}0^\circ$) measured in the range $-14 \leq h \leq 14$, $-19 \leq k \leq 19$, $-13 \leq l \leq 1$. No significant

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