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## Structure Reports

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# Monoclinic modification of 1,1,3,3,5,5-hexamethyl-cyclo-1,3,5-tristannathiane

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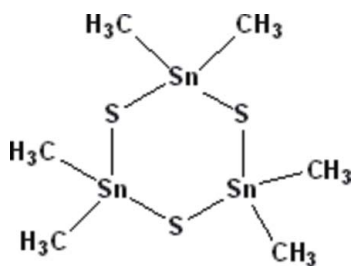
Received 10 November 2007; accepted 27 November 2007

 Key indicators: single-crystal X-ray study;  $T = 150$  K; mean  $\sigma(\text{Sn}-\text{C}) = 0.004$  Å;  $R$  factor = 0.030;  $wR$  factor = 0.067; data-to-parameter ratio = 40.7.

The asymmetric unit of the title compound,  $[\text{Sn}_3(\text{CH}_3)_6\text{S}_3]$ , contains two molecules with twist-boat conformations. There are intermolecular  $\text{S}\cdots\text{H}$  (2.929 Å),  $\text{S}\cdots\text{S}$  (3.433 Å),  $\text{S}\cdots\text{C}$  (3.465 Å) and  $\text{C}\cdots\text{H}$  (2.898 Å) interactions in addition to prominent intermolecular  $\text{Sn}\cdots\text{S}$  interactions of 3.692 and 3.769 Å.

## Related literature

For related literature, see: Menzebach & Bleckmann (1975) (tetragonal form); Jacobsen & Krebs (1977) (monoclinic form); Farina *et al.* (2001) (tetragonal form); Spek (2003).



## Experimental

### Crystal data

$[\text{Sn}_3(\text{CH}_3)_6\text{S}_3]$	$V = 3192.94$ (4) Å <sup>3</sup>
$M_r = 542.45$	$Z = 8$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 14.826$ (1) Å	$\mu = 5.01$ mm <sup>-1</sup>
$b = 12.814$ (1) Å	$T = 150$ (2) K
$c = 17.744$ (1) Å	$0.25 \times 0.25 \times 0.20$ mm
$\beta = 108.706$ (1)°	

### Data collection

Nonius KappaCCD diffractometer	78537 measured reflections
Absorption correction: multi-scan (SORTAV; Blessing 1995)	9326 independent reflections
$T_{\min} = 0.311$ , $T_{\max} = 0.361$	8221 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.046$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$	229 parameters
$wR(F^2) = 0.067$	H-atom parameters constrained
$S = 1.15$	$\Delta\rho_{\text{max}} = 0.93$ e Å <sup>-3</sup>
9326 reflections	$\Delta\rho_{\text{min}} = -1.69$ e Å <sup>-3</sup>

Data collection: *COLLECT* (Nonius, 1997–2000); cell refinement: *HKL* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2203).

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**supplementary materials**

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## Monoclinic modification of 1,1,3,3,5,5-hexamethyl-*cyclo*-1,3,5-tristannathiane

N. Singh, A. Kumar, K. C. Molloy and G. Kociok-Köhn

### Comment

Tris(dimethyltin sulfide), 1,1,3,3,5,5-hexamethyl-*cyclo*-1,3,5-tristannathiane was the unexpected product in our attempt to synthesizing dimethyltin(emda) (emda = 1-ethoxycarbonyl-1-methylcarbonyl-2,2-dithiolate) (see *Experimental*). The literature reports that the compound crystallizes in monoclinic ( $P2_1/c$ ; Jacobsen & Krebs, 1977), tetragonal ( $P_4$ ; Menzebach & Bleckmann, 1975) and tetragonal ( $P4_22_12$ ; Farina *et al.*, 2001) modifications. The monoclinic modification was refined in the  $P2_1/c$  space group. However, the checking program *PLATON* (Spek, 2003) finds  $P2_1/n$  space group which is now being authenticated in the present study. In the monoclinic unit cell the molecules are linked by Sn $\cdots$ S interaction of 3.692 and 3.796 Å, S $\cdots$ H interaction of 2.929 Å, S $\cdots$ S interaction of 3.433 Å, S $\cdots$ C interaction of 3.465 Å and C $\cdots$ H interaction of 2.898 Å.

### Experimental

To a stirring 20 ml methanolic solution of  $K_2emda$  (1 mmol) was added, 15 ml methanolic solution of dimethyltin(IV) chloride (1 mmol). The mixture was additionally stirred for 2 h. Whole solvent was vacuum evaporated to obtain solid residue. To this 20 ml chloroform was added and suction filtered to discard KCl. The orange coloured solution thus obtained was layered with methanol to afford yellow crystals.

### Figures



Fig. 1. ORTEP plot of tris(dimethyltin sulfide) at the 30% probability level.

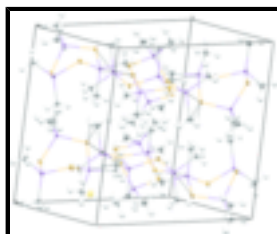


Fig. 2. Unit cell packing of tris(dimethyltin sulfide) showing Sn $\cdots$ S, S $\cdots$ S, S $\cdots$ H, S $\cdots$ C and C $\cdots$ H interactions.

## 1,1,3,3,5,5-hexamethyl-*cyclo*-1,3,5-tristannathiane

### Crystal data

[Sn<sub>3</sub>(CH<sub>3</sub>)<sub>6</sub>S<sub>3</sub>]

$M_r = 542.45$

Monoclinic,  $P2_1/n$

$D_x = 2.257 \text{ Mg m}^{-3}$

Melting point: 148 K

Mo  $K\alpha$  radiation

$\lambda = 0.71073 \text{ \AA}$

# supplementary materials

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$a = 14.826 (1) \text{ \AA}$   
 $b = 12.814 (1) \text{ \AA}$   
 $c = 17.744 (1) \text{ \AA}$   
 $\beta = 108.706 (1)^\circ$   
 $V = 3192.94 (4) \text{ \AA}^3$   
 $Z = 8$   
 $F_{000} = 2016$

Cell parameters from 24000 reflections  
 $\theta = 2.9\text{--}27.5^\circ$   
 $\mu = 5.01 \text{ mm}^{-1}$   
 $T = 150 (2) \text{ K}$   
Block, yellow  
 $0.25 \times 0.25 \times 0.20 \text{ mm}$

## Data collection

Nonius KappaCCD diffractometer	9326 independent reflections
Radiation source: fine-focus sealed tube	8221 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.046$
$T = 150(2) \text{ K}$	$\theta_{\text{max}} = 30.1^\circ$
363 1.6 degree images with $\varphi$ and $\omega$ scans	$\theta_{\text{min}} = 3.2^\circ$
Absorption correction: multi-scan (SORTAV; Blessing 1995)	$h = -19 \rightarrow 20$
$T_{\text{min}} = 0.311$ , $T_{\text{max}} = 0.361$	$k = -17 \rightarrow 18$
78537 measured reflections	$l = -24 \rightarrow 24$

## Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.030$	H-atom parameters constrained
$wR(F^2) = 0.067$	$w = 1/[\sigma^2(F_o^2) + (0.0287P)^2 + 3.7599P]$
$S = 1.15$	where $P = (F_o^2 + 2F_c^2)/3$
9326 reflections	$(\Delta/\sigma)_{\text{max}} = 0.002$
229 parameters	$\Delta\rho_{\text{max}} = 0.93 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -1.69 \text{ e \AA}^{-3}$
	Extinction correction: none

## Special details

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > 2\sigma(F^2)$  is used only for calculating  $R$ -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Sn1	0.407801 (14)	-0.393311 (15)	0.330468 (12)	0.02220 (5)
Sn2	0.292630 (16)	-0.244657 (15)	0.143631 (12)	0.02433 (5)
Sn3	0.260380 (14)	-0.539580 (15)	0.143724 (12)	0.02231 (5)
Sn4	0.595000 (14)	0.105237 (15)	0.215469 (13)	0.02279 (5)
Sn5	0.775307 (14)	0.255910 (15)	0.373674 (12)	0.02168 (5)
Sn6	0.784610 (16)	-0.045519 (16)	0.370081 (13)	0.02807 (5)
S1	0.38690 (6)	-0.21710 (6)	0.28053 (5)	0.02717 (15)
S2	0.34811 (6)	-0.40429 (6)	0.10117 (5)	0.02742 (16)
S3	0.26038 (5)	-0.48569 (6)	0.27553 (4)	0.02501 (14)
S4	0.73866 (6)	0.00869 (6)	0.23204 (5)	0.02779 (15)
S5	0.74693 (8)	0.10168 (6)	0.44061 (5)	0.03455 (19)
S6	0.63617 (6)	0.28120 (6)	0.25940 (5)	0.02786 (15)
C1	0.5255 (3)	-0.4688 (3)	0.3101 (3)	0.0405 (8)
H1A	0.5840	-0.4305	0.3374	0.061*
H1B	0.5160	-0.4705	0.2528	0.061*
H1C	0.5306	-0.5403	0.3306	0.061*
C2	0.4245 (3)	-0.3748 (3)	0.4546 (2)	0.0427 (9)
H2A	0.4120	-0.4415	0.4764	0.064*
H2B	0.3794	-0.3222	0.4609	0.064*
H2C	0.4896	-0.3522	0.4832	0.064*
C3	0.1441 (2)	-0.2544 (3)	0.1283 (2)	0.0361 (8)
H3A	0.1203	-0.1853	0.1360	0.054*
H3B	0.1340	-0.3032	0.1673	0.054*
H3C	0.1100	-0.2793	0.0744	0.054*
C4	0.3309 (3)	-0.1254 (3)	0.0757 (2)	0.0411 (9)
H4A	0.2846	-0.1239	0.0220	0.062*
H4B	0.3945	-0.1398	0.0724	0.062*
H4C	0.3313	-0.0577	0.1014	0.062*
C5	0.1159 (2)	-0.5364 (3)	0.06916 (19)	0.0285 (6)
H5A	0.0969	-0.6062	0.0472	0.043*
H5B	0.1090	-0.4869	0.0256	0.043*
H5C	0.0753	-0.5146	0.1003	0.043*
C6	0.3447 (2)	-0.6776 (2)	0.1555 (2)	0.0298 (6)
H6A	0.3090	-0.7375	0.1655	0.045*
H6B	0.4038	-0.6693	0.2000	0.045*
H6C	0.3598	-0.6892	0.1063	0.045*
C7	0.5300 (3)	0.1155 (3)	0.0895 (2)	0.0423 (9)
H7A	0.5087	0.0461	0.0680	0.063*
H7B	0.5763	0.1425	0.0655	0.063*
H7C	0.4751	0.1627	0.0774	0.063*
C8	0.5078 (3)	0.0398 (3)	0.2777 (3)	0.0429 (9)
H8A	0.4453	0.0737	0.2603	0.064*
H8B	0.5379	0.0506	0.3350	0.064*
H8C	0.5001	-0.0352	0.2665	0.064*
C9	0.7751 (3)	0.3803 (2)	0.4535 (2)	0.0313 (7)

## supplementary materials

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H9A	0.7892	0.3526	0.5075	0.047*
H9B	0.7123	0.4138	0.4371	0.047*
H9C	0.8236	0.4318	0.4525	0.047*
C10	0.9028 (3)	0.2408 (3)	0.3437 (2)	0.0364 (8)
H10A	0.9176	0.3075	0.3233	0.055*
H10B	0.8942	0.1869	0.3027	0.055*
H10C	0.9554	0.2209	0.3912	0.055*
C11	0.6965 (3)	-0.1716 (3)	0.3834 (2)	0.0416 (9)
H11A	0.7122	-0.2347	0.3589	0.062*
H11B	0.6295	-0.1537	0.3573	0.062*
H11C	0.7075	-0.1843	0.4401	0.062*
C12	0.9340 (3)	-0.0702 (3)	0.4128 (2)	0.0425 (9)
H12A	0.9538	-0.0860	0.4698	0.064*
H12B	0.9667	-0.0072	0.4040	0.064*
H12C	0.9503	-0.1289	0.3842	0.064*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Sn1	0.02077 (10)	0.02012 (10)	0.02404 (10)	0.00078 (7)	0.00487 (8)	0.00072 (7)
Sn2	0.02967 (11)	0.01939 (10)	0.02465 (10)	0.00053 (7)	0.00974 (8)	0.00356 (7)
Sn3	0.02227 (10)	0.01899 (9)	0.02482 (10)	0.00015 (7)	0.00638 (8)	-0.00101 (7)
Sn4	0.02074 (10)	0.02090 (10)	0.02697 (11)	-0.00286 (7)	0.00798 (8)	-0.00433 (7)
Sn5	0.02387 (10)	0.01951 (10)	0.02113 (10)	0.00017 (7)	0.00645 (8)	-0.00146 (7)
Sn6	0.03327 (12)	0.01932 (10)	0.02936 (11)	-0.00007 (8)	0.00690 (9)	0.00062 (8)
S1	0.0324 (4)	0.0176 (3)	0.0293 (4)	-0.0025 (3)	0.0070 (3)	-0.0022 (3)
S2	0.0326 (4)	0.0234 (4)	0.0311 (4)	-0.0020 (3)	0.0171 (3)	-0.0019 (3)
S3	0.0253 (3)	0.0250 (4)	0.0266 (3)	-0.0048 (3)	0.0110 (3)	-0.0019 (3)
S4	0.0304 (4)	0.0268 (4)	0.0287 (4)	0.0056 (3)	0.0129 (3)	-0.0016 (3)
S5	0.0580 (6)	0.0244 (4)	0.0275 (4)	0.0035 (3)	0.0225 (4)	0.0037 (3)
S6	0.0296 (4)	0.0178 (3)	0.0299 (4)	0.0010 (3)	0.0008 (3)	0.0002 (3)
C1	0.0286 (17)	0.040 (2)	0.054 (2)	0.0095 (15)	0.0137 (16)	0.0019 (17)
C2	0.053 (2)	0.048 (2)	0.0230 (16)	-0.0027 (18)	0.0071 (16)	-0.0010 (15)
C3	0.0268 (17)	0.0341 (18)	0.043 (2)	0.0044 (13)	0.0058 (15)	-0.0018 (14)
C4	0.064 (3)	0.0281 (17)	0.040 (2)	0.0004 (17)	0.0280 (19)	0.0102 (14)
C5	0.0243 (14)	0.0335 (16)	0.0260 (15)	0.0020 (12)	0.0056 (12)	0.0023 (12)
C6	0.0322 (16)	0.0234 (14)	0.0351 (16)	0.0051 (12)	0.0126 (13)	0.0015 (12)
C7	0.038 (2)	0.050 (2)	0.0300 (18)	0.0039 (16)	-0.0020 (15)	-0.0081 (15)
C8	0.0358 (19)	0.042 (2)	0.059 (2)	-0.0133 (16)	0.0270 (18)	-0.0004 (17)
C9	0.0403 (18)	0.0248 (15)	0.0263 (15)	0.0008 (13)	0.0071 (14)	-0.0085 (12)
C10	0.0282 (17)	0.0370 (18)	0.049 (2)	0.0013 (13)	0.0187 (15)	0.0021 (15)
C11	0.051 (2)	0.0274 (17)	0.041 (2)	-0.0122 (16)	0.0082 (17)	0.0065 (14)
C12	0.0344 (19)	0.046 (2)	0.039 (2)	0.0020 (16)	-0.0001 (15)	-0.0160 (17)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

Sn1—C1	2.125 (3)	C3—H3A	0.9800
Sn1—C2	2.149 (4)	C3—H3B	0.9800
Sn1—S3	2.4005 (8)	C3—H3C	0.9800

Sn1—S1	2.4090 (8)	C4—H4A	0.9800
Sn2—C4	2.133 (3)	C4—H4B	0.9800
Sn2—C3	2.133 (4)	C4—H4C	0.9800
Sn2—S1	2.4086 (8)	C5—H5A	0.9800
Sn2—S2	2.4136 (8)	C5—H5B	0.9800
Sn3—C5	2.126 (3)	C5—H5C	0.9800
Sn3—C6	2.136 (3)	C6—H6A	0.9800
Sn3—S2	2.4284 (8)	C6—H6B	0.9800
Sn3—S3	2.4386 (8)	C6—H6C	0.9800
Sn4—C8	2.123 (3)	C7—H7A	0.9800
Sn4—C7	2.135 (4)	C7—H7B	0.9800
Sn4—S4	2.3982 (8)	C7—H7C	0.9800
Sn4—S6	2.3999 (8)	C8—H8A	0.9800
Sn5—C10	2.131 (3)	C8—H8B	0.9800
Sn5—C9	2.133 (3)	C8—H8C	0.9800
Sn5—S6	2.4060 (8)	C9—H9A	0.9800
Sn5—S5	2.4112 (8)	C9—H9B	0.9800
Sn6—C12	2.121 (4)	C9—H9C	0.9800
Sn6—C11	2.138 (3)	C10—H10A	0.9800
Sn6—S4	2.4240 (8)	C10—H10B	0.9800
Sn6—S5	2.4259 (8)	C10—H10C	0.9800
C1—H1A	0.9800	C11—H11A	0.9800
C1—H1B	0.9800	C11—H11B	0.9800
C1—H1C	0.9800	C11—H11C	0.9800
C2—H2A	0.9800	C12—H12A	0.9800
C2—H2B	0.9800	C12—H12B	0.9800
C2—H2C	0.9800	C12—H12C	0.9800
C1—Sn1—C2	113.19 (17)	Sn2—C3—H3C	109.5
C1—Sn1—S3	113.37 (11)	H3A—C3—H3C	109.5
C2—Sn1—S3	105.13 (12)	H3B—C3—H3C	109.5
C1—Sn1—S1	112.33 (11)	Sn2—C4—H4A	109.5
C2—Sn1—S1	103.44 (12)	Sn2—C4—H4B	109.5
S3—Sn1—S1	108.66 (3)	H4A—C4—H4B	109.5
C4—Sn2—C3	114.88 (16)	Sn2—C4—H4C	109.5
C4—Sn2—S1	106.94 (12)	H4A—C4—H4C	109.5
C3—Sn2—S1	112.25 (11)	H4B—C4—H4C	109.5
C4—Sn2—S2	104.57 (11)	Sn3—C5—H5A	109.5
C3—Sn2—S2	110.58 (10)	Sn3—C5—H5B	109.5
S1—Sn2—S2	107.06 (3)	H5A—C5—H5B	109.5
C5—Sn3—C6	121.18 (13)	Sn3—C5—H5C	109.5
C5—Sn3—S2	109.01 (9)	H5A—C5—H5C	109.5
C6—Sn3—S2	105.44 (9)	H5B—C5—H5C	109.5
C5—Sn3—S3	106.35 (9)	Sn3—C6—H6A	109.5
C6—Sn3—S3	108.70 (9)	Sn3—C6—H6B	109.5
S2—Sn3—S3	105.12 (3)	H6A—C6—H6B	109.5
C8—Sn4—C7	115.01 (17)	Sn3—C6—H6C	109.5
C8—Sn4—S4	113.41 (12)	H6A—C6—H6C	109.5
C7—Sn4—S4	104.21 (11)	H6B—C6—H6C	109.5
C8—Sn4—S6	109.64 (11)	Sn4—C7—H7A	109.5

## supplementary materials

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C7—Sn4—S6	105.33 (11)	Sn4—C7—H7B	109.5
S4—Sn4—S6	108.75 (3)	H7A—C7—H7B	109.5
C10—Sn5—C9	115.11 (15)	Sn4—C7—H7C	109.5
C10—Sn5—S6	113.04 (11)	H7A—C7—H7C	109.5
C9—Sn5—S6	105.97 (10)	H7B—C7—H7C	109.5
C10—Sn5—S5	110.93 (10)	Sn4—C8—H8A	109.5
C9—Sn5—S5	104.36 (10)	Sn4—C8—H8B	109.5
S6—Sn5—S5	106.72 (3)	H8A—C8—H8B	109.5
C12—Sn6—C11	116.90 (17)	Sn4—C8—H8C	109.5
C12—Sn6—S4	109.05 (12)	H8A—C8—H8C	109.5
C11—Sn6—S4	110.17 (11)	H8B—C8—H8C	109.5
C12—Sn6—S5	108.57 (10)	Sn5—C9—H9A	109.5
C11—Sn6—S5	106.29 (12)	Sn5—C9—H9B	109.5
S4—Sn6—S5	105.18 (3)	H9A—C9—H9B	109.5
Sn2—S1—Sn1	101.43 (3)	Sn5—C9—H9C	109.5
Sn2—S2—Sn3	103.76 (3)	H9A—C9—H9C	109.5
Sn1—S3—Sn3	104.42 (3)	H9B—C9—H9C	109.5
Sn4—S4—Sn6	102.86 (3)	Sn5—C10—H10A	109.5
Sn5—S5—Sn6	106.13 (3)	Sn5—C10—H10B	109.5
Sn4—S6—Sn5	101.96 (3)	H10A—C10—H10B	109.5
Sn1—C1—H1A	109.5	Sn5—C10—H10C	109.5
Sn1—C1—H1B	109.5	H10A—C10—H10C	109.5
H1A—C1—H1B	109.5	H10B—C10—H10C	109.5
Sn1—C1—H1C	109.5	Sn6—C11—H11A	109.5
H1A—C1—H1C	109.5	Sn6—C11—H11B	109.5
H1B—C1—H1C	109.5	H11A—C11—H11B	109.5
Sn1—C2—H2A	109.5	Sn6—C11—H11C	109.5
Sn1—C2—H2B	109.5	H11A—C11—H11C	109.5
H2A—C2—H2B	109.5	H11B—C11—H11C	109.5
Sn1—C2—H2C	109.5	Sn6—C12—H12A	109.5
H2A—C2—H2C	109.5	Sn6—C12—H12B	109.5
H2B—C2—H2C	109.5	H12A—C12—H12B	109.5
Sn2—C3—H3A	109.5	Sn6—C12—H12C	109.5
Sn2—C3—H3B	109.5	H12A—C12—H12C	109.5
H3A—C3—H3B	109.5	H12B—C12—H12C	109.5

Fig. 1

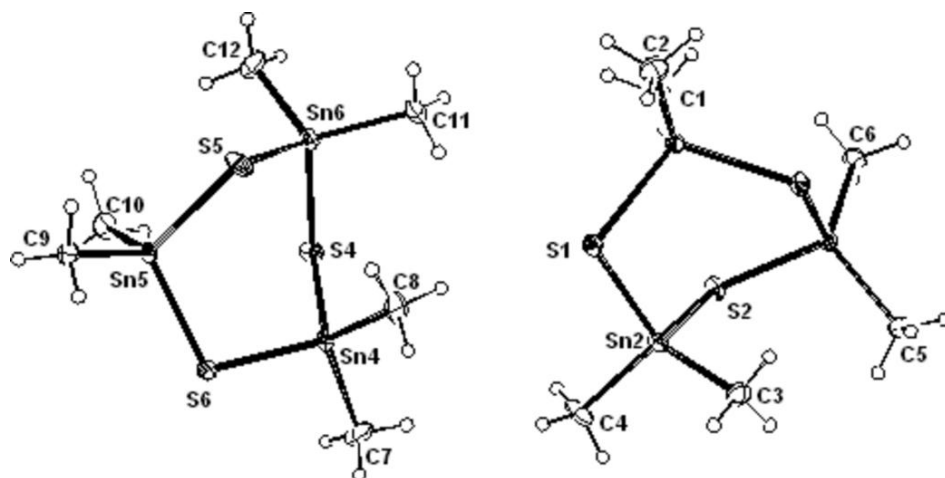


Fig. 2

