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Tetragonal modification of 1,1,3,3,5,5-hexamethylcyclo-1,3,5-tristannathiane

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(S-C) = 0.008 Å
R factor = 0.037
wR factor = 0.080
Data-to-parameter ratio = 33.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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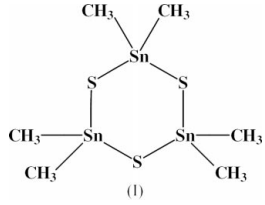
The space group of the title compound, $[Sn_3S_3(CH_3)_6]$, reported as $P4_1$, is corrected to $P4_22_12$.

Comment

Tris(dimethyltin sulfide), 1,1,3,3,5,5-hexamethylcyclo-1,3,5-tristannatiane, (I), was the unexpected product in our attempt at synthesizing dimethyltin bis(dipropyldithiocarbamate) from dimethyltin dichloride, carbon disulfide and *n*-propylamine (see *Experimental*). The literature reports that the compound crystallizes in monoclinic ($P2_1/c$; Jacobsen & Krebs, 1977) and tetragonal (Menzebach & Bleckmann, 1975) modifications. The tetragonal modification was refined in the $P4_1$ space group; however, the checking program *PLATON* (Spek, 1990) finds missing symmetry elements, and the $P4_22_12$ space group is suggested. This space group is now authenticated in the present study. The two independent Sn atoms in the tetragonal modification exist in tetrahedral geometries; adjacent molecules are linked by an Sn···S interaction of 3.902 (2) Å into a linear chain.



(I)

Experimental

A solution of carbon disulfide (5 mmol) in methanol (5 ml) was added to a methanolic solution (20 ml) of a mixture of dimethyltin(IV) chloride (2.5 mmol) and *n*-propylamine (5 mmol). The reagent solutions were cooled to 273 K, and the carbon disulfide solution was then added at a rate so that the temperature of the mixture did not rise above 278 K. The mixture was stirred at 275 K for 2 h. A cream-colored solid was formed that was collected by filtration and washed with cold methanol (2 × 5 ml). The product was recrystallized from a methanol/chloroform (3:2 *v*/*v*) mixture to afford colorless crystals.

Crystal data

$[Sn_3S_3(CH_3)_6]$
M_r = 542.45
Tetragonal, $P4_12_12$
a = 9.7249 (1) Å
c = 17.3867 (3) Å
V = 1644.32 (4) Å^3
Z = 4
D_x = 2.191 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5984 reflections
θ = 2.4–28.2°
μ = 4.87 mm^{-1}
T = 298 (2) K
Block, colorless
0.22 × 0.14 × 0.08 mm

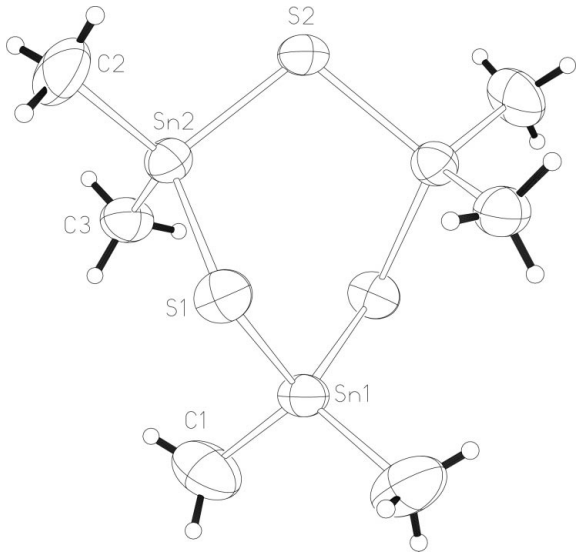


Figure 1
*ORTEP*II (Johnson, 1976) plot of tris(dimethyltin sulfide) at the 50% probability level. H atoms are shown as circles of arbitrary radii.

Data collection

Siemens CCD area-detector diffractometer	1995 independent reflections
ω scans	1545 reflections with $I > 2\sigma(I)$
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)	$R_{int} = 0.088$
$T_{min} = 0.414$, $T_{max} = 0.697$	$\theta_{max} = 28.2^\circ$
11 469 measured reflections	$h = -9 \rightarrow 12$
	$k = -12 \rightarrow 12$
	$l = -22 \rightarrow 22$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0368P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.037$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.080$	$\Delta\rho_{max} = 0.50$ e Å^{-3}
$S = 0.94$	$\Delta\rho_{min} = -0.86$ e Å^{-3}
1995 reflections	Absolute structure: Flack & Schwarzenbach (1988)
59 parameters	Flack parameter = 0.08 (7)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Sn1—C1	2.125 (8)	Sn1—S1	2.406 (2)
Sn2—C2	2.134 (7)	Sn2—S1	2.435 (2)
Sn2—C3	2.126 (7)	Sn2—S2	2.424 (2)
C1—Sn1—C1^{i}	113.9 (5)	C2—Sn2—S2	104.0 (3)
C1—Sn1—S1	111.9 (3)	C3—Sn2—S1	108.2 (2)
C1—Sn1—S1^{i}	105.3 (3)	C3—Sn2—S2	112.2 (2)
S1—Sn1—S1^{i}	108.6 (1)	S1—Sn2—S2	107.1 (1)
C2—Sn2—C3	119.1 (3)	Sn1—S1—Sn2	103.1 (1)
C2—Sn2—S1	105.5 (2)	Sn2—S2—Sn2^{i}	103.1 (1)

Symmetry code: (i) $y, x, -z$.

Of the 1995 reflections, 1216 were unique and 779 were Friedel pairs.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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