

## 2,5-Bis[(cyclopentylphenylstannyl)sulfanyl]-1,3,4-thiadiazole

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The two Sn atoms in the title compound,  $[\text{Sn}_2(\text{C}_5\text{H}_9)_2(\text{C}_6\text{H}_5)_4(\text{C}_2\text{N}_2\text{S}_3)]$ , exist in tetrahedral geometries.

Received 21 April 2004

Accepted 27 April 2004

Online 8 May 2004

## Key indicators

Single-crystal X-ray study

 $T = 298 \text{ K}$ Mean  $\sigma(\text{N}-\text{C}) = 0.011 \text{ \AA}$ 

Disorder in main residue

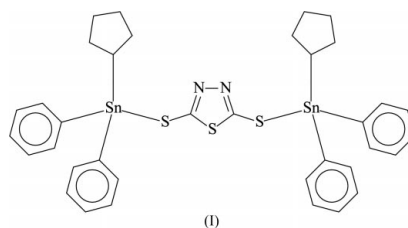
 $R$  factor = 0.031 $wR$  factor = 0.071

Data-to-parameter ratio = 8.8

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

## Comment

The Sn atom in triorganotin mercaptides generally exists in a tetrahedral geometry, as noted in, for example, triphenyltin 2-aminophenylsulfide (Ng *et al.*, 1989) and 5-amino-1,3,4-thiadiazolyl-2-thiolate (Ng *et al.*, 1990). The dimercaptan, 2,5-dimercapto-1,3,4-thiadiazole, which has been used to bind to other metal complexes (Tannai *et al.*, 2003), is used to bind to two mixed alkyl/aryl tin(IV) cations in the title complex, (I). The study of this mixed organotin system continues from a previous study of cyclopentylphenyltin compounds, which were synthesized (Ong *et al.*, 1993) to examine the influence of the cycloalkyl unit on the biological properties of the resulting triorganotin compound (Nazni *et al.*, 1994).



The molecular structure of (I), is shown in Fig. 1, and it can be seen that the two Sn atoms exist in tetrahedral geometries. Selected bond distances and angles are given in Table 1. The Sn–S bond length is similar to that found in 5-amino-1,3,4-thiadiazolyl-2-thiolate (Ng *et al.*, 1990).

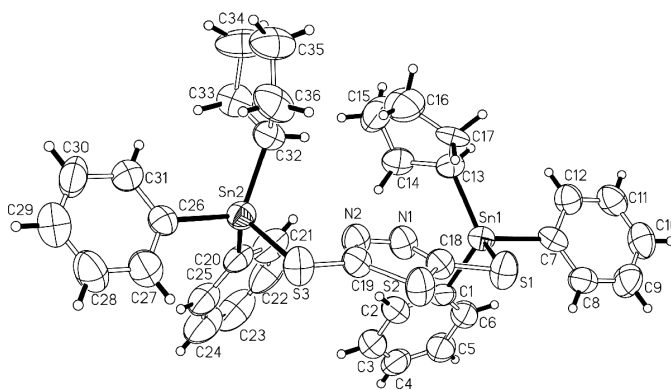


Figure 1

ORTEP (Johnson, 1976) plot of the title complex, (I), showing the numbering scheme and displacement ellipsoids at the 50% probability level. Only one of the disordered components is shown.

## Experimental

2,5-Dimercapto-1,3,4-thiadiazole was purchased from Fluka. Cyclopentylidiphenyltin hydroxide was synthesized by a multi-step reaction, starting from the Grignard reaction of cyclopentylmagnesium bromide and triphenyltin chloride. One of the phenyl groups was cleaved by using iodine in DMF; the resulting iodide was then exchanged with a hydroxide unit (Lo *et al.*, 1999). The mercaptan (0.4 g, 0.27 mmol) and the triorganotin hydroxide (1.9 g, 0.54 mmol) were dissolved in hot ethanol. The solution was filtered; colorless crystals separated when the solution was cooled slowly. CH&N analysis for C<sub>36</sub>H<sub>38</sub>N<sub>2</sub>S<sub>3</sub>Sn<sub>2</sub>: C 50.8, H 4.88, N 3.13%; calculated: C 51.8, H 4.56, N 3.36%.

### Crystal data

[Sn <sub>2</sub> (C <sub>5</sub> H <sub>9</sub> ) <sub>2</sub> (C <sub>6</sub> H <sub>5</sub> ) <sub>4</sub> (C <sub>2</sub> N <sub>2</sub> S <sub>3</sub> )]	$D_x = 1.561 \text{ Mg m}^{-3}$
$M_r = 832.24$	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 25 reflections
$a = 9.968 (1) \text{ \AA}$	$\theta = 12.0\text{--}14.0^\circ$
$b = 18.381 (2) \text{ \AA}$	$\mu = 1.61 \text{ mm}^{-1}$
$c = 10.215 (1) \text{ \AA}$	$T = 298 (2) \text{ K}$
$\beta = 108.883 (6)^\circ$	Block, colorless
$V = 1771.0 (3) \text{ \AA}^3$	$0.3 \times 0.3 \times 0.2 \text{ mm}$
$Z = 2$	

### Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.016$
$\omega$ - $2\theta$ scans	$\theta_{\text{max}} = 25.0^\circ$
Absorption correction: $\psi$ scan (North <i>et al.</i> , 1968)	$h = -11 \rightarrow 0$
$T_{\text{min}} = 0.648$ , $T_{\text{max}} = 0.724$	$k = -21 \rightarrow 0$
3400 measured reflections	$l = -11 \rightarrow 12$
3207 independent reflections	3 standard reflections
2788 reflections with $I > 2\sigma(I)$	frequency: 60 min
	intensity decay: 2%

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0413P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.031$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.071$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 0.97$	$\Delta\rho_{\text{max}} = 0.30 \text{ e \AA}^{-3}$
3207 reflections	$\Delta\rho_{\text{min}} = -0.26 \text{ e \AA}^{-3}$
365 parameters	Absolute structure: Flack (1983), no
H-atom parameters constrained	Friedel pairs
	Flack parameter = 0.00 (7)

**Table 1**

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

Sn1—C1	2.137 (4)	Sn2—C20	2.124 (4)
Sn1—C7	2.142 (5)	Sn2—C26	2.141 (5)
Sn1—C13	2.138 (6)	Sn2—C32	2.119 (7)
Sn1—S1	2.481 (2)	Sn2—S3	2.473 (2)
C1—Sn1—C7	105.9 (2)	C20—Sn2—C26	109.2 (2)
C1—Sn1—C13	116.5 (2)	C20—Sn2—C32	117.8 (3)
C1—Sn1—S1	107.6 (2)	C20—Sn2—S3	109.3 (2)
C7—Sn1—C13	110.6 (2)	C26—Sn2—C32	111.5 (3)
C7—Sn1—S1	98.7 (2)	C26—Sn2—S3	99.1 (2)
C13—Sn1—S1	115.7 (2)	C32—Sn2—S3	108.3 (2)

The structure is disordered in respect of the two cyclopentyl rings and the disorder affects other parts of the molecule. The phenyl rings were refined as rigid hexagons ( $\text{C—C} = 1.39 \text{ \AA}$ ). The cyclopentyl rings are disordered over two positions, but the occupancy could not be refined. Instead the disorder was regarded as 50:50. The C—C distances were restrained to  $1.50 (1) \text{ \AA}$ , and the 1,3-related C··C distances to  $2.45 (1) \text{ \AA}$ . The displacement parameters of the unprimed and primed atoms were made equal to each other. The C14/C14' pair of atoms could not be refined anisotropically without restraints; the atoms were restrained to be approximately isotropic. The H atoms were placed at calculated positions [phenyl C—H =  $0.93 \text{ \AA}$  and  $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$ ; methylene C—H =  $0.97 \text{ \AA}$  and  $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$ ; methine C—H =  $0.98 \text{ \AA}$  and  $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$ ], and were included in the refinement in the riding-model approximation.

Data collection: CAD-4/PC (Kretschmar, 1994); cell refinement: CAD-4 VAX/PC Fortran System (Enraf–Nonius, 1988); data reduction: XCAD4 (Harms, 1997) in WinGX (Farrugia, 1999); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEPII (Johnson, 1976); software used to prepare material for publication: SHELXL97.

The authors thank the Ministry of Science, Technology & the Environment (IRPA 09-02-03-0100 EA100) and the University of Malaya for supporting this work.

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