

## A 1:1 adduct between 2,2-bis(chlorodimethylstannyl)propane and dimethyl sulfoxide

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## Key indicators

Single-crystal X-ray study

$T = 291\text{ K}$

Mean  $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$

Disorder in solvent or counterion

$R$  factor = 0.026

w $R$  factor = 0.050

Data-to-parameter ratio = 35.5

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

In the title compound,  $[\text{Sn}_2(\text{CH}_3)_4(\text{C}_3\text{H}_6)\text{Cl}_2] \cdot \text{C}_2\text{H}_6\text{OS}$ , the single dimethyl sulfoxide (DMSO) molecule bridges the two Sn atoms *via* its O atom [Sn—O distances: 2.578 (2) and 2.632 (2) Å], so that each Sn atom displays distorted trigonal-bipyramidal geometry. The S atom is disordered over two positions with occupancy factors 0.596 (2) and 0.404 (2).

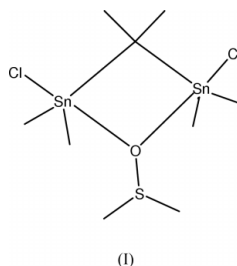
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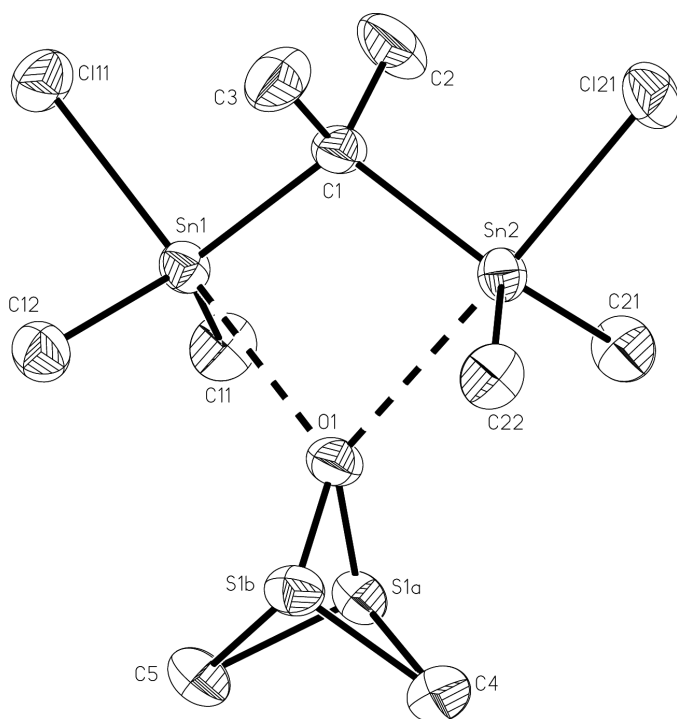
## Comment

Organotin compounds of the type  $R_3\text{Sn}X$ , where  $X$  is an electronegative group, such as halide or trifluoromethanesulfonate, form complexes with donor ligands  $L$  in which the Sn atom becomes pentacoordinate. It might thus be expected that compounds of the type  $X\text{Sn}R_2(\text{CR}'R'')_n\text{Sn}R_2X$  would form similar complexes in which a ligand  $L$  is attached to each Sn atom. However, a study involving DMSO as ligand (Karol *et al.*, 1983) shows that the compound  $\text{ClSnMe}_2\text{CH}_2\text{SnMeCl}_2$  forms a 1:1 complex with DMSO, which bridges the two Sn atoms *via* its O atom, (I).



It thus seemed of interest to determine whether the replacement of the H atoms of the  $\text{CH}_2$  group between the Sn atoms by methyl groups would cause the complex to adopt another geometry. Also of interest are variations in the Sn—C—Sn angle, since previous NMR work (Mitchell *et al.*, 1983) indicates that the coupling constant  $^2J(\text{Sn}-\text{C}-\text{Sn})$  changes sign at an angle very close to the tetrahedral angle of  $109.47^\circ$ .

The present results show that 1:1 complexation of DMSO is present in the title compound (I) and that its geometry is broadly similar to that of  $\text{ClSnMe}_2\text{CH}_2\text{SnMeCl}_2 \cdot \text{DMSO}$ , (II). There are, however, significant differences. While in (II) the angle Sn—C—Sn is  $112.0(6)^\circ$ , it decreases in (I) to  $110.39(13)^\circ$ . The Sn— $\text{CH}_2$  bond lengths are, of course, considerably different in (II):  $\text{ClMe}_2\text{Sn}-\text{CH}_2$  2.159 (3) and  $\text{Cl}_2\text{MeSn}-\text{CH}_2$  2.097 (13) Å. In (I), the two bond lengths are, as expected, almost identical: 2.164 (3) and 2.152 (3) Å. The angle Sn—O—Sn is very similar in (I) and (II):  $85.69(6)$  and



**Figure 1**  
View of the title compound showing the labelling of all non-H atoms. Displacement ellipsoids are shown at 30% probability levels. The S atom is disordered [two positions with occupancy factors: 0.596 (2) and 0.404 (2)].

86.6 (2)°, respectively. However, the Sn—O bond distances differ considerably: 2.578 (2) and 2.632 (2) Å in (I), and 2.568 (8) and 2.575 (8) Å in (II).

## Experimental

0.3 g (0.73 mmol) of 2,2-bis(chlorodimethylstannyl)propane (Austin *et al.*, 1986; Karol *et al.*, 1983) were dissolved in 2 ml of dry dimethyl sulfoxide and the mixture stirred for 30 min. The solution was left overnight at 278–288 K. The crystals were separated and dried carefully to remove the solvent from their surface; m.p. 391–392 K, yield 60% [literature (Austin *et al.*, 1987) 389–390 K].

### Crystal data

[Sn<sub>2</sub>(CH<sub>3</sub>)<sub>4</sub>(C<sub>3</sub>H<sub>6</sub>)Cl<sub>2</sub>]·C<sub>2</sub>H<sub>6</sub>OS  
 $M_r = 488.62$   
 Monoclinic,  $P2_1/c$   
 $a = 10.7602$  (2) Å  
 $b = 9.1559$  (2) Å  
 $c = 18.2953$  (3) Å  
 $\beta = 99.2514$  (12)°  
 $V = 1779.00$  (6) Å<sup>3</sup>  
 $Z = 4$

$D_x = 1.824$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 14885 reflections  
 $\theta = 3.0$ – $32.0^\circ$   
 $\mu = 3.21$  mm<sup>-1</sup>  
 $T = 291$  (1) K  
 Block, colourless  
 0.44 × 0.42 × 0.40 mm

### Data collection

Nonius KappaCCD diffractometer  
 258 frames via  $\omega$ -rotation ( $\Delta\omega = 1^\circ$ )  
 and two times 15 s per frame  
 (three sets at different  $\kappa$ -angles)  
 Absorption correction: none  
 14885 measured reflections  
 5357 independent reflections

3091 reflections with  $I > 2\sigma(I)$   
 $R_{int} = 0.026$   
 $\theta_{max} = 32.0^\circ$   
 $h = -15 \rightarrow 15$   
 $k = -10 \rightarrow 13$   
 $l = -26 \rightarrow 26$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.026$   
 $wR(F^2) = 0.050$   
 $S = 0.86$   
 5357 reflections  
 151 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0169P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} = 0.002$   
 $\Delta\rho_{max} = 0.40$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -0.45$  e Å<sup>-3</sup>  
 Extinction correction: *SHELXL97*  
 Extinction coefficient: 0.0230 (3)

Two positions with occupancy factors of 0.596 (2) and 0.404 (2) for S1A and S1B, respectively, were refined for the S atom of the dimethyl sulfoxide moiety. H atoms were placed in calculated positions with  $U_{iso}$  constrained to be 1.5 times  $U_{eq}$  of the carrier atom. For the methyl groups containing atoms C11, C12, C21 and C22, the torsion angles were refined, whereas for the remaining methyl groups AFIX 33 (Sheldrick, 1997) was used. At C4 and at C5 there are two different orientations of the methyl groups having the same site occupation factors as the corresponding positions of the S atom.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*, *PARST95* (Nardelli, 1995) and *PLATON* (Spek, 2001).

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