

Moumita Sen Sarma,^a Carol A. Ellis,^b Nirmalya Moitra,^a Abhijit Roy^{a‡} and Edward R. T. Tiekink^{b*}^aDepartment of Chemistry, North Bengal University, Dt. Darjeeling, West Bengal 734 013, India, and ^bDepartment of Chemistry, The University of Texas at San Antonio, One UTSA Circle, San Antonio, Texas 78249-0698, USA‡ Correspondence e-mail:
abhijitchem1947@yahoo.co.inCorrespondence e-mail:
edward.tiekink@utsa.edu

Key indicators

Single-crystal X-ray study
T = 173 K
Mean $\sigma(\text{C}-\text{C})$ = 0.008 Å
R factor = 0.050
wR factor = 0.117
Data-to-parameter ratio = 17.9For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.Di- μ_2 -methoxy-bis[benzyl(5-chloro-2-oxido-benzaldehyde thiosemicarbazonato)tin(IV)]

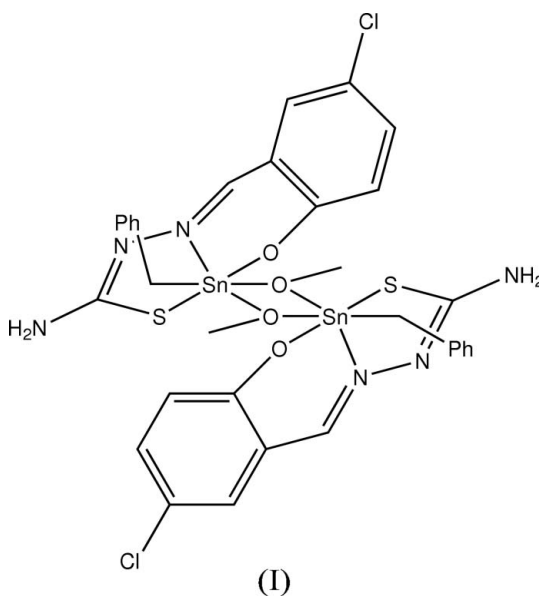
The centrosymmetric title compound, $[\text{Sn}_2(\text{C}_7\text{H}_7)_2(\text{C}_8\text{H}_6\text{ClN}_3\text{OS})_2(\text{CH}_3\text{O})_2]$, displays octahedrally coordinated tin within a CNO_3S donor set. Molecules aggregate *via* $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds, leading to a chain, and these bonds combined with $\text{N}-\text{H}\cdots\pi$ and $\text{C}-\text{H}\cdots\pi$ interactions lead to a three-dimensional array.

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Comment

Interest in compounds related to the title compound stems from their potential biological activity (Farrell, 2002; Gielen & Tiekink, 2005). The title organotin thiosemicarbazone, (I), was isolated as yellow prisms from the attempted recrystallization of an authenticated sample of $(\text{PhCH}_2)_2\text{SnL}$, where LH_2 is *p*-chlorosalicylaldehydethiosemicarbazone, from a methanol solution. The crystal structure analysis shows a centrosymmetric molecule (Fig. 1) in which two $(\text{PhCH}_2)\text{Sn}$ entities are symmetrically bridged by two methoxide ligands (Table 1). The distorted octahedral coordination geometry for Sn is completed by N-, O- and S-donor atoms derived from the dinegative and tridentate L^{2-} ligand. Distortions from ideal geometry may be traced to the strain found in the centrosymmetric Sn_2O_2 core and chelate rings.



Molecules are held in a three-dimensional array by a combination of $\text{N}-\text{H}\cdots\text{N}$, $\text{N}-\text{H}\cdots\pi$ and $\text{C}-\text{H}\cdots\pi$ interactions. The hydrogen bonding between $\text{N1}-\text{H1A}$ and N2^{ii} [symmetry code: (ii) $1-x, -y, 1-z$] leads to the formation of chains along $[101]$ (see Fig. 2). The parameters associated with

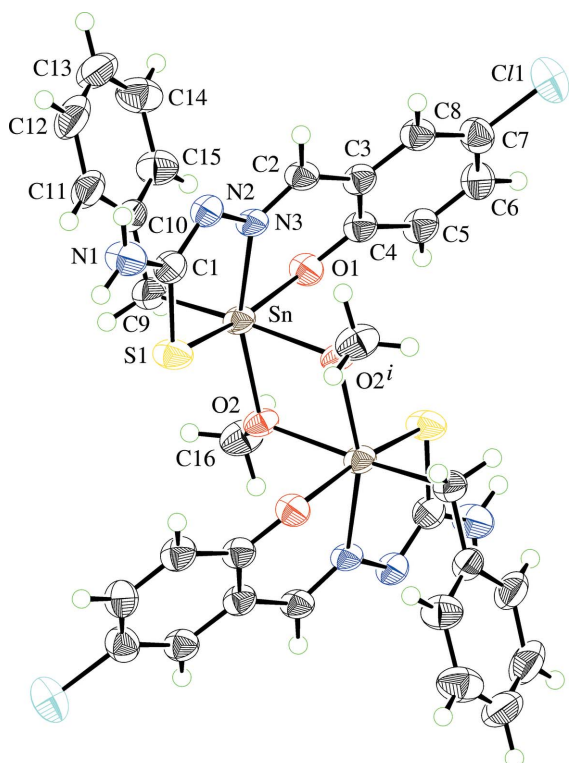


Figure 1
The molecular structure and atom-labelling scheme for (I), showing 50% probability displacement ellipsoids. [Symmetry code: (i) $-x, -y, -z$.]

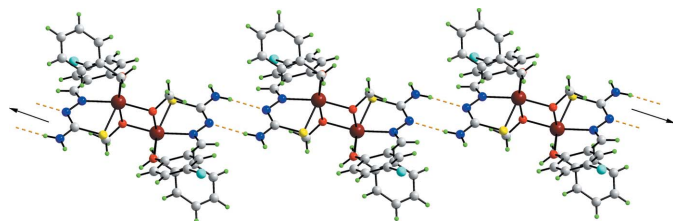


Figure 2
Chains mediated by N—H...H hydrogen-bonding interactions, shown as golden dashed lines, in (I) (Crystal Impact, 2006). Colour code: Sn (brown), Cl (cyan), S (yellow), O (red), N (blue), C (grey) and H (green).

these interactions are $N1-H1A \cdots N2^{ii} = 2.26 \text{ \AA}$ and $N1 \cdots N2^{ii} = 3.126(6) \text{ \AA}$ with an angle of 169° at H1A. While not involved in a conventional hydrogen-bonding interaction, the amine atom H1B forms an N—H... π interaction with the ring centroid of the aromatic portion of a tin-bound benzyl residue so that the $N1-H1B \cdots$ ring centroid (C10–C15)ⁱⁱⁱ distance is 2.81 \AA with an angle of 176° at H1B [symmetry code: (iii) $1 - x, -\frac{1}{2} + y, \frac{1}{2} - z$]; some of these interactions are highlighted with an '(a)' in Fig. 3. The methoxide-bound methyl-H atoms form C—H... π interactions with the six-membered aromatic ring (C3–C8)^{iv} so that the H...ring centroid distance is 2.66 \AA and the angle at H is 122° [symmetry code (iv) $x, \frac{1}{2} - y, -\frac{1}{2} + z$], shown as '(b)' in Fig. 3.

Experimental

The compound was isolated as a decomposition product of a sample of $(C_6H_5CH_2)_2SnL_2$ during attempted recrystallization from MeOH

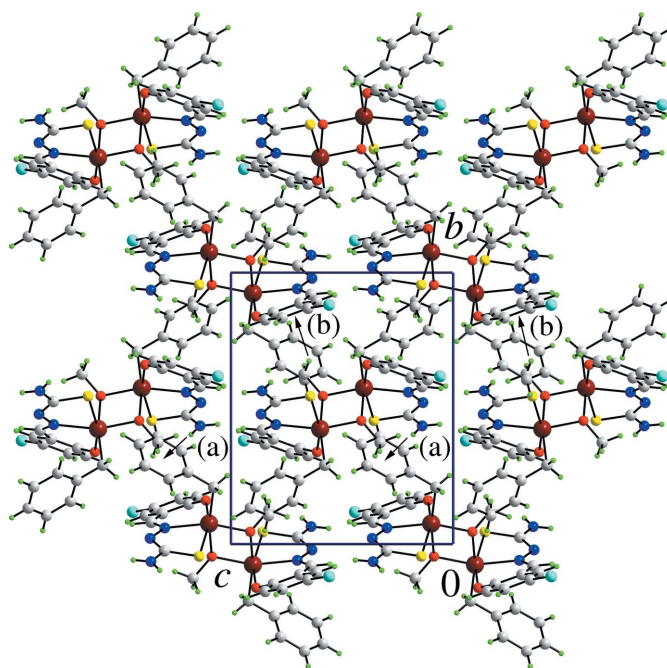


Figure 3
The packing of (I), viewed down the *a* axis (Crystal Impact, 2006). Colour code as in Fig. 2.

solution (m.p. 417–419 K). A methanol solution (100 ml) of *p*-chlorosalicylaldehydethiosemicarbazone (0.500 g, 2.17 mmol), prepared by a literature procedure (Basuli *et al.*, 1998), was stirred continuously in a 0.1 N methanol NaOH solution (45.9 ml, 0.174 g, 4.35 mmol) for 2 h. $(C_6H_5CH_2)_2SnCl_2$ (0.809 g, 2.17 mmol), prepared by a literature procedure (Sisido *et al.*, 1961), was then added to the reaction mixture which was refluxed for 6 h under an inert atmosphere. The volatiles were removed by distillation and the crude product obtained was washed thoroughly with hot petroleum ether (b.p. 333–353 K) in quantities of 4–5 ml, extracted into benzene (50 ml) and filtered. The yellow crystalline product was obtained upon repeated crystallization from the benzene extract. Yield 30%. IR (Bruker Equinox SS; cm^{-1}): $\nu(NH_2)$ 3285, $\nu(C=N)_{azo}$ 1619, $\nu(C=N)_{thia}$ 1596, $\nu(C-O)$ 1184, $\nu(C-S)$ 996, and $\nu(C-S-N)$ 754. The TGA (Shimadzu thermogravimetric analyser) showed a distinct weight loss between 412 and 439 K corresponding to loss of two OMe fragments. Subsequent weight loss occurred, resulting in the clean formation of SnS beyond 895 K.

Crystal data

$[Sn_2(C_7H_7)_2(C_8H_6ClN_3OS)_2 \cdot (CH_3O)_2]$	$V = 1729.1(13) \text{ \AA}^3$
$M_r = 937.04$	$Z = 2$
Monoclinic, $P2_1/c$	$D_x = 1.800 \text{ Mg m}^{-3}$
$a = 11.617(7) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 13.484(4) \text{ \AA}$	$\mu = 1.77 \text{ mm}^{-1}$
$c = 12.430(4) \text{ \AA}$	$T = 173(2) \text{ K}$
$\beta = 117.367(5)^\circ$	Prism, pale yellow
	$0.15 \times 0.05 \times 0.05 \text{ mm}$

Data collection

Rigaku AFC12K/SATURN724 diffractometer	44284 measured reflections
ω scans	3906 independent reflections
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	3887 reflections with $I > 2\sigma(I)$
$T_{min} = 0.813, T_{max} = 1$ (expected range = 0.744–0.915)	$R_{int} = 0.051$
	$\theta_{max} = 27.6^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.117$
 $S = 1.22$
 3906 reflections
 218 parameters
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0396P)^2 + 5.0883P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.99 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.77 \text{ e } \text{\AA}^{-3}$

Table 1

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

Sn—S1	2.4945 (16)	O1—C4	1.326 (6)
Sn—N3	2.221 (4)	O2—C16	1.430 (6)
Sn—O1	2.045 (3)	N1—C1	1.343 (6)
Sn—O2	2.108 (3)	N2—N3	1.388 (5)
Sn—C9	2.168 (5)	N2—C1	1.317 (6)
Sn—O2 ⁱ	2.164 (3)	N3—C2	1.289 (6)
S1—C1	1.753 (5)		
S1—Sn—O1	162.51 (9)	O2—Sn—O2 ⁱ	72.46 (13)
S1—Sn—O2	99.97 (10)	C9—Sn—O2 ⁱ	167.90 (16)
S1—Sn—N3	78.35 (10)	C9—Sn—N3	103.09 (16)
S1—Sn—C9	99.41 (15)	N3—Sn—O2 ⁱ	86.36 (13)
S1—Sn—O2 ⁱ	89.82 (10)	Sn—S1—C1	94.97 (18)
O1—Sn—O2	93.59 (13)	Sn—O1—C4	126.4 (3)
O1—Sn—N3	85.00 (13)	Sn—O2—C16	118.9 (3)
O1—Sn—C9	89.49 (17)	Sn ⁱ —O2—C16	123.0 (3)
O1—Sn—O2 ⁱ	83.75 (14)	Sn—O2—Sn ⁱ	107.54 (13)
O2—Sn—N3	158.79 (14)	Sn—N3—N2	119.9 (3)
O2—Sn—C9	98.05 (15)	Sn—N3—C2	124.3 (3)

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

H atoms were included in the riding-model approximation with C—H = 0.95 \AA to 0.99 \AA , and N—H = 0.88 \AA , and with $U_{\text{iso}}(\text{H}) =$

$1.5U_{\text{eq}}(\text{C})$ for methyl, and $1.2U_{\text{eq}}(\text{C,N})$ for the remaining H atoms.

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *PATY* in *DIRDIF92* (Beurskens *et al.*, 1992); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976) and *DIAMOND* (Crystal Impact, 2006); software used to prepare material for publication: *SHELXL97*.

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