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Novel organotin(IV) compounds derived from bis(organostannyl)methanes: Synthesis and crystal structures of bis[diphenyl(pyridin-2-onato)stannyl]methane and bis[bromophenyl(pyrimidine-2-thionato)stannyl]methane · C_7H_8

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Abstract

Reaction between bis(chlorodiphenylstanyl)methane and the sodium salt of 2-hydroxypyridine (pyONa) in the molar ratio of 1:2 provides the organotin hydroxide derivative $[Ph_2(pyO)SnCH_2Sn(OH)Ph_2]_2$ (**1**) (where pyO = anion of 2-hydroxypyridine), while reaction of bis(dibromophenylstanyl)methane with the sodium salt of pyrimidine-2-thione (pmtNa) in molar ratio of 1:4 gives the corresponding organotin thiolate derivative, as its toluene solvate $[BrPh(pmt)Sn]_2CH_2 \cdot C_7H_8$ (**2**) (where pmt = anion of pyrimidine-2-thione). Both compounds were characterized by single crystal X-ray diffraction analysis and contain five-coordinate tin atoms. Compound **1** is a centrosymmetric head-to-tail dimmer with almost symmetrical Sn(1)–O(H)–Sn(2A) bridges.

Keywords: Organotin compounds; 2-Hydroxypyridine; Pyrimidine-2-thione; X-ray diffraction analysis

1. Introduction

As result of their widespread applications, organotin compounds have attracted great interest over many decades [1,2]. The most important applications are probably those related to the catalytical activity of tetraorganodistannoxanes in many organic reactions [3] and their use as industrial and agricultural biocides because of their antifungal properties [4,5]. For some time diorganotin compounds are being investigated for their anti-tumor activity [6]. The interest in the chemistry of heterocyclic thiones arises from their wide range of applications in analytical chemistry, in medicine and as biocides [7]. Especially pyrimidine-2-thione shows bacteriostatic activity [8] while it has been found to inhibit the synthesis of t-RNA [9]. A number of organotin compounds with thiolates has been synthesized and studied for their biocidal [10] and antitumour [11] activities, while their catalytic activity in regio- and stereoselective syntheses of γ-diketones as well as α,β-dihydroxy ketones [12,13] have also been reported. Although many organotin compounds containing a single tin atom with pyrimidine-2-thiolate or pyridine-2-thiolate moieties and their derivatives have been structurally characterized [14–26], there are very few reports on the synthesis of organotin compounds containing more than one tin atom and thioles [25–27]. On the other hand, pyridinone-based ligands have been studied in view of their use as controlling reagents for the level of metals in body and in clinical diagnosis and chemotherapy [28,29]. Surprisingly there are very few reports on the structural characterization of organotin(IV) compounds containing 2-hydroxypyridine

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[30a] while, the synthesis of dibutyltin(IV) derivative of 2-hydroxypyrine has been also reported recently [30b].

Here we report the synthesis and structural characterization of the novel bis(organostannyl)methane derivatives $[Ph_2(pyO)SnCH_2Sn(OH)Ph_2]_2$ (**1**) (pyO = the anion of 2-hydroxypyridine) and $[BrPh(pmt)Sn]_2CH_2 \cdot C_7H_8$ (**2**) (pmt = anion of pyrimidine-2-thione).

2. Experimental

2.1. Materials and instruments

All solvents used were reagent grade, while 2-hydroxypyridine and pyrimidine-2-thione (Fluka, Aldrich) were used without further purification. The bis(haloorganostannyl)methanes $[(ClPh_2Sn)_2CH_2]$ and $[(Br_2PhSn)_2CH_2]$ were prepared as described previously [27]. Elemental analyses for C, H and N were carried out with an instrument Carlo Erba Strumentazione, model 1106. The melting points were measured in open tubes with a Buchi SMP-20 apparatus and are uncorrected. Infra-red spectra in the region of 4000–370 cm^{-1} were obtained with a Bruker IFS 113v spectrometer from samples pressed in KBr discs. A Bruker DPX-400 spectrometer was used for obtaining ^1H NMR spectra.

2.2. Synthesis of bis[(μ-hydroxo)(pyridin-2-onato){bis(diphenyl-stanyl)methane}] (1)

A suspension of 2-hydroxypyridine (pyOH) (0.231 g, 2.43 mmol) in distilled water (1 cm^{-3}) was treated with a one-molar solution of NaOH (2.43 mL, 2,43 mmol). The resulting clear, pale yellow solution was stirred while a solution of $(ClPh_2Sn)_2CH_2$ (0.769 g, 1.22 mmol) in methanol (1 mL) was added. A colourless powder precipitated. It was filtered, washed with 3 mL of cold distilled water and dried in vacuo over silica gel. Recrystallization of the solid material by slow evaporation of its toluene/dichloromethane solution provided colourless single crystals of (**1**) suitable for X-ray diffraction. M.p. = 61–63 °C. Elemental analysis found: C, 53.65 H, 4.15; N, 2.45%, calculated for $C_{60}H_{54}N_2O_4Sn_4$: C, 53.71; H, 4.05; N, 2.09%. IR (cm^{-1}): 3450br, 3045m, 2911w, 1640m, 1609m, 1571w, 1480vs, 1429vs, 1332w, 1302w, 1261w, 1190w, 1156w, 1076vs, 997m, 911w, 728vs, 698vs, 660m, 590s, 446s. ^1H NMR ($CDCl_3$) chemical shifts δ (ppm): 7.46–7.16 (m, 40H, Ph and 8H of pyO), 1.65 (br, 2H, OH), 0.81, $^2J(^{119}Sn–C–^1H)$ = 54 Hz (s, 4H, $SnCH_2$).

2.3. Preparation of the bis[(bromophenyl(pyrimidine-2-thionato)stanyl)]methane · C_7H_8 (2)

A suspension of pyrimidine-2-thione (pmtH) (0.224 g, 2 mmol) in distilled water (1 mL) was treated with a one-molar solution of NaOH (2 mL, 2 mmol). The resulting clear yellow solution was stirred while a solution of $(Br_2PhSn)_2CH_2$ (0.363 g, 0.5 mmol) in methanol (1 mL) was added. A colourless powder precipitated. It was filtered, washed with 3 mL of cold distilled water and dried in vacuo over silica gel. Recrystallization of the solid material by slow evaporation of its toluene/dichloromethane solution provided pale yellow single crystals of (**2**) suitable for X-ray diffraction. M.p. = 125 °C. Elemental analysis found: C, 38.4; H, 2.9; N, 5.8%, calculated for $Br_2C_{28}H_{26}N_4S_2Sn_2$: C, 38.22; H, 2.98; N, 6.37%. IR (cm^{-1}): 3056w, 2917w, 1560vs, 1547vs, 1426s, 1383vs, 1250s, 1182vs, 799s, 735vs, 696vs, 466s, 270s. ^1H NMR ($CDCl_3$) chemical shifts δ (ppm): 8.5 (d, 2H, N=CH(6)– of pmt), 8.4 (d, 2H, CH(4)–N– of pmt), 7.6 (m, 4H, Ph), 7.3 (m, 6H, Ph), 7.1 (m, 5H, toluene), 7.0 (t, 2H –CH(5)–C=N of pmt), 2.3 (s, 3H, toluene), 1.98, $^2J(^{119}Sn–C–^1H)$ = 72 Hz (s, 2H, $SnCH_2$).

2.4. X-ray data collection and reduction of the intensity data of the molecules (1) and (2)

Intensity data for the colourless (**1**) and the light yellow (**2**) crystals were collected on a Nonius Kappa CCD diffractometer with graphite-monochromated MoKα radiation. The data collection for molecule **1** covered almost the whole sphere of reciprocal space with two sets at different κ-angles and 205 frames via ω-rotation ($\Delta/\omega = 1°$) at two times 10 s for **1** per frame while in case of molecule **2** the data collection covered almost the whole sphere of reciprocal space with three sets at different κ-angles and 293 frames via ω-rotation ($\Delta/\omega = 1°$) at two times 10 s for **2** per frame. The crystal-to-detector distances were 3.4 cm for both molecules. Crystal decay was monitored by repeating the initial frames at the end of data collection. Analysing the duplicate reflections there was no indication for any decay. The structures was solved by direct methods SHELXS97 [31] and successive difference Fourier syntheses. Refinement applied full-matrix least-squares methods SHELXL97 [32]. The H atoms were placed in geometrically calculated positions using a riding model (including free rotation about Sn–O in case of **1**) with U_{iso} constrained at 1.2 for non-methyl groups and 1.5 for methyl groups times U_{eq} of the carrier C atom. Atomic scattering factors for neutral atoms and real and imaginary dispersion terms were taken from *International Tables for X-ray Crystallography* [33]. The figures were created by SHELXTL [34]. Crystallographic data for molecules **1** and **2** are given in Table 1, while selected bond distances and angles in Tables 2 and 3, respectively. Thermal ellipsoid representations of the molecules together with atomic numbering schemes are shown in Figs. 1 and 3.

3. Results and discussion

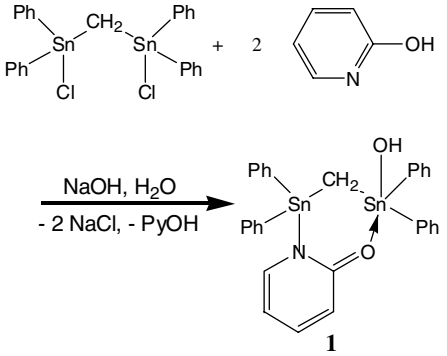
3.1. Synthesis

The reaction of bis(chlorodiphenylstannyl)methane with an aqueous solution of 2-hydroxypyridine and sodium hydroxide gave the monosubstituted organotin compound

Table 1
Crystal data and the structure refinement details for the molecules **1** and **2**

Compound	**1**	**2**
Empirical formula	$C_{60}H_{54}N_2O_4Sn_4$	$C_{28}H_{26}Br_2N_4S_2Sn_2$
Formula weight	1341.81	879.85
Temperature (K)	173(1)	173(1)
Wavelength (Å)	0.71073	0.71073
Crystal system, space group	Monoclinic, $P21/c$	Monoclinic, $C2/c$
Unit cell dimensions		
a (Å)	11.4494(3)	8.8430(3)
b (Å)	10.9443(3)	16.9106(4)
c (Å)	21.0907(5)	21.4249(6)
β (°)	97.6422(17)	99.6049(16)
Volume (Å^3)	2619.31(12)	3158.98(16)
Z	2	4
Density (calculated) (Mg/m^3)	1.701	1.850
Density (measured)	Not determined	Not determined
Absorption coefficient (mm^{-1})	1.934	4.267
F(000)	1320	1696
Crystal size (mm)	$0.18 \times 0.12 \times 0.10$	$0.18 \times 0.10 \times 0.08$
θ Range for data collection (°)	3.09–27.48	3.09–27.48.
Index ranges	$-14 \leqslant h \leqslant 14$, $-14 \leqslant h \leqslant 14$, $-27 \leqslant h \leqslant 27$	$-11 \leqslant h \leqslant 11$, $-21 \leqslant k \leqslant 21$, $-27 \leqslant l \leqslant 27$
Reflections collected/ unique [R_{int}]	16906/5930 [0.0410]	14087/3598 [0.0730]
Reflections observed [$I > 2$sigma(I)]	3911	2450
Completeness to $2\theta = 27.48$	98.7%	98.9%
Absorption correction	None	None
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/restraints/parameters	5930/0/316	3598/0/177
Goodness-of-fit on F^2	0.900	0.960
Final R indices [$I > 2$sigma(I)]	$R_1 = 0.0339$, $wR_2 = 0.0571$	$R_1 = 0.0378$, $wR_2 = 0.0801$
R indices (all data)	$R_1 = 0.0655$, $wR_2 = 0.0606$	$R_1 = 0.0697$, $wR_2 = 0.0859$
Maximum shift/e.s.d.	0.001	0.001
Largest difference in peak and hole (e Å^{-3})	0.901 and −0.754	1.219 and −0.901

1 in which the second chloride anion is replaced by hydroxide.



(1)

1

Reaction of bis(dibromophenylstannyl)methane with excess of the sodium salt of pyrimidine-2-thione gave compound **2** with only twofold substitution of the Sn–Br functions.

Table 2
Selected bond lengths (Å) and angles (°) for the molecule **1** with e.s.d.'s in parentheses

(a) Bond lengths (Å)			
Sn(1)–C(21)	2.127(3)	Sn(2)–N(51)	2.354(3)
Sn(1)–C(1)	2.133(3)	O(51)–C(56)	1.287(4)
Sn(1)–C(11)	2.143(4)	N(51)–C(56)	1.348(4)
Sn(1)–O(1)	2.2038(19)	N(51)–C(52)	1.367(4)
Sn(1)–O(51)	2.227(2)	C(52)–C(53)	1.355(5)
Sn(2)–C(1)	2.126(3)	C(53)–C(54)	1.392(5)
Sn(2)–C(31)	2.132(3)	C(54)–C(55)	1.369(5)
Sn(2)–C(41)	2.140(3)	C(55)–C(56)	1.411(5)
Sn(2)–O(1A)	2.261(2)		
(b) Angles (°)			
C(21)–Sn(1)–C(1)	125.60(12)	C(1)–Sn(2)–C(41)	118.56(13)
C(21)–Sn(1)–C(11)	115.04(14)	C(31)–Sn(2)–C(41)	112.38(12)
C(1)–Sn(1)–C(11)	118.43(14)	C(1)–Sn(2)–O(1A)	89.08(11)
C(21)–Sn(1)–O(1)	93.05(10)	C(31)–Sn(2)–O(1A)	89.59(11)
C(1)–Sn(1)–O(1)	92.82(10)	C(41)–Sn(2)–O(1A)	92.67(10)
C(11)–Sn(1)–O(1)	93.77(10)	C(1)–Sn(2)–N(51)	88.45(11)
C(21)–Sn(1)–O(51)	87.01(10)	C(31)–Sn(2)–N(51)	91.41(11)
C(1)–Sn(1)–O(51)	85.67(10)	C(41)–Sn(2)–N(51)	89.15(11)
C(11)–Sn(1)–O(51)	87.85(11)	O(1A)–Sn(2)–N(51)	177.42(8)
O(1)–Sn(1)–O(51)	178.18(8)	Sn(1)–O(1)–Sn(2A)	136.31(10)
C(1)–Sn(2)–C(31)	129.05(12)	C(56)–O(51)–Sn(1)	133.8(2)

Symmetry transformations used to generate equivalent atoms: #1 $-x+1$, $-y+1$, $-z$.

Table 3
Selected bond lengths (Å) and angles (°) for the molecule **2** with e.s.d.'s in parentheses

(a) Bond lengths (Å)			
Sn(1)–C(11)	2.120(4)	C(7)–Sn(1)#1	2.128(3)
Sn(1)–C(7)	2.128(3)	C(11)–C(12)	1.385(6)
Sn(1)–S(1)	2.4277(11)	C(11)–C(16)	1.387(5)
Sn(1)–N(2)	2.476(3)	C(12)–C(13)	1.381(6)
Sn(1)–Br(1)	2.5535(6)	C(13)–C(14)	1.381(7)
S(1)–C(1)	1.748(5)	C(14)–C(15)	1.364(7)
N(2)–C(3)	1.325(5)	C(15)–C(16)	1.382(6)
N(2)–C(1)	1.358(5)	C(21)–C(22)	1.345(9)
N(6)–C(5)	1.322(6)	C(21)–C(23)#2	1.412(9)
N(6)–C(1)	1.327(5)	C(22)–C(23)	1.397(9)
C(3)–C(4)	1.375(6)	C(23)–C(24)	1.285(12)
C(4)–C(5)	1.376(6)	C(23)–C(21)#2	1.412(9)
(b) Angles (°)			
C(11)–Sn(1)–C(7)	116.25(16)	N(2)–C(3)–C(4)	121.6(4)
C(11)–Sn(1)–S(1)	113.78(11)	C(3)–C(4)–C(5)	116.4(4)
C(7)–Sn(1)–S(1)	123.31(14)	N(6)–C(5)–C(4)	124.3(4)
C(11)–Sn(1)–N(2)	90.96(13)	Sn(1)#1–C(7)–Sn(1)	119.5(3)
C(7)–Sn(1)–N(2)	89.90(11)	C(12)–C(11)–C(16)	119.3(4)
S(1)–Sn(1)–N(2)	64.19(8)	C(12)–C(11)–Sn(1)	119.3(3)
C(11)–Sn(1)–Br(1)	101.77(11)	C(16)–C(11)–Sn(1)	121.4(3)
C(7)–Sn(1)–Br(1)	101.23(4)	C(13)–C(12)–C(11)	120.3(4)
S(1)–Sn(1)–Br(1)	93.00(3)	C(12)–C(13)–C(14)	119.5(5)
N(2)–Sn(1)–Br(1)	156.99(8)	C(15)–C(14)–C(13)	120.9(4)
C(1)–S(1)–Sn(1)	87.37(13)	C(14)–C(15)–C(16)	119.8(4)
C(3)–N(2)–C(1)	116.8(4)	C(15)–C(16)–C(11)	120.3(4)
C(3)–N(2)–Sn(1)	148.2(3)	C(22)–C(21)–C(23)#2	120.4(7)
C(1)–N(2)–Sn(1)	95.0(2)	C(21)–C(22)–C(23)	120.8(7)
C(5)–N(6)–C(1)	115.0(4)	C(24)–C(23)–C(22)	121.6(9)
N(6)–C(1)–N(2)	125.9(4)	C(24)–C(23)–C(21)#2	119.5(9)
N(6)–C(1)–S(1)	120.7(3)	C(22)–C(23)–C(21)#2	118.8(7)
N(2)–C(1)–S(1)	113.4(3)		

Symmetry transformations used to generate equivalent atoms: #1 $-x$, y, $-z+3/2$; #2 $-x-1$, $-y$, $-z+1$.

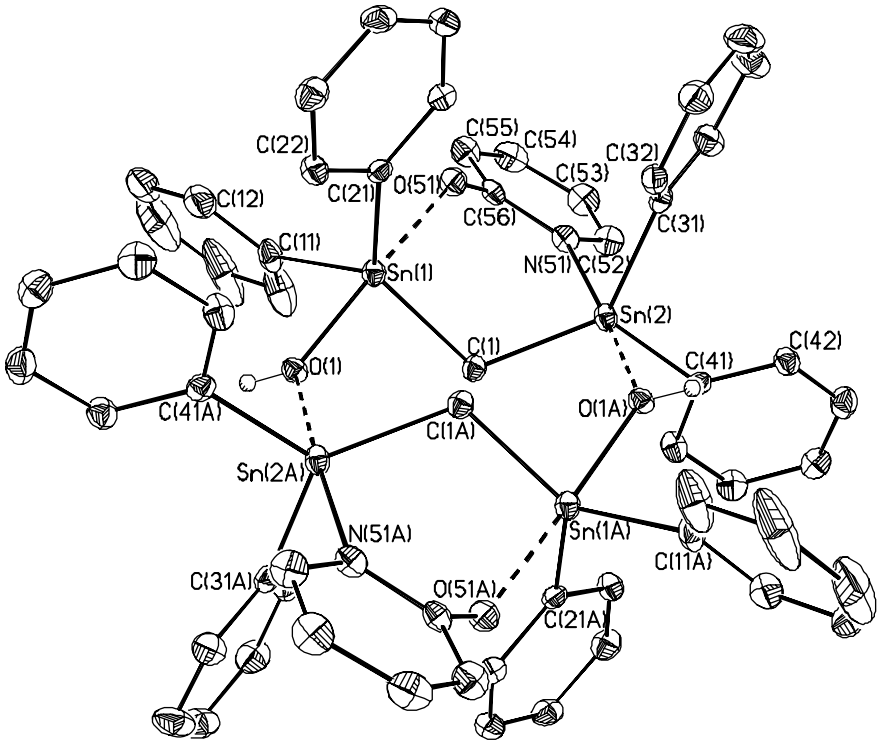
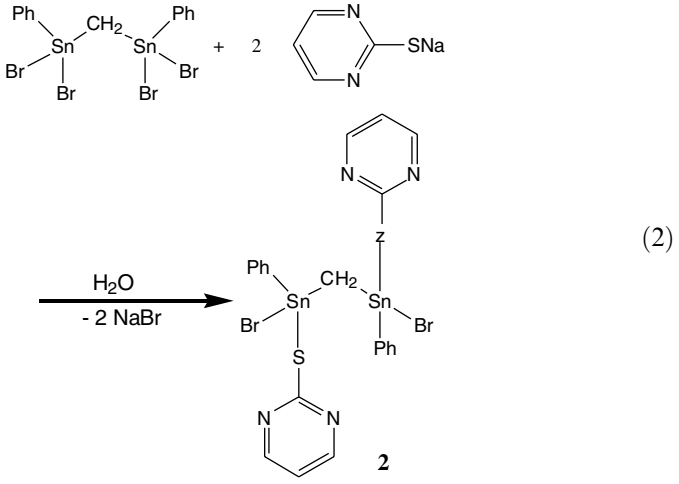


Fig. 1. General view (SHELXTL) of a molecule of 1 showing 30% probability displacement ellipsoids and the atom numbering scheme.



(2)

Both compounds are crystalline solids and well soluble in common organic solvents such as dichloromethane, chloroform, benzene, toluene etc.

3.2. Molecular structure of compound *1*

A view of a molecule of **1** is shown in Fig. 1 and selected geometric parameters are given in Table 2. Compound **1** forms a centrosymmetric head-to-tail dimer via an almost symmetrical intermolecular Sn–O(H) ··· Sn bridge of 2.204(2) and 2.261(2) Å. Both the Sn(1) and Sn(2) atom show each a distorted trigonal bipyramidal configuration (geometrical goodness $\Delta(\Sigma\vartheta)$ [35,36] 79.4° (Sn1), 88.7° (Sn2)) with the C(1), C(11), C(21) and C(1), C(31), C(41) carbon atoms occupying the equatorial positions at Sn(1) and Sn(2), respectively. The axial positions are occupied by O(1) and O(51) at Sn(1) and O(1A) and N(51) at Sn(2). The Sn(1)–O(1) and Sn(1)–O(51) distances of 2.204(2) and 2.227(2) Å are almost equal whereas the Sn(2)–O(1A) distance is slightly expanded to 2.261(2) Å as result of the nitrogen donor atom in *trans* position with a N(51)–Sn(2) distance of 2.354(3) Å.

The O(1)–Sn(1)–C(1)–Sn(2)–O(1A)–Sn(1A)–C(1A)–Sn(2A)– eight membered ring is not planar and adopts a D-type [35,36] conformation (Fig. 2). The deviation from the least square Sn(1)C(1)Sn(1A)C(1A) plane is for Sn(2) = 1.9384(0.0045), for O(1) = −1.6264(0.0031), for Sn(2A) = −1.9384(0.0045) and for O(1) = 1.6264(0.0031) Å, respectively, while the O(1)–Sn(1)–C(1)–Sn(2) torsion angle is −138.47(15)°.

3.3. Molecular structure of compound *2*

A view of the molecule **2** is shown in Fig. 3, while selected bond distances and angles are given in Table 3. The molecule is essentially monomeric with the shortest intermolecular distances being beyond the sum of the van

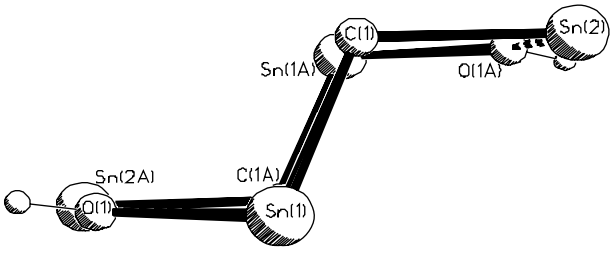


Fig. 2. Classification scheme for the eight member ring.

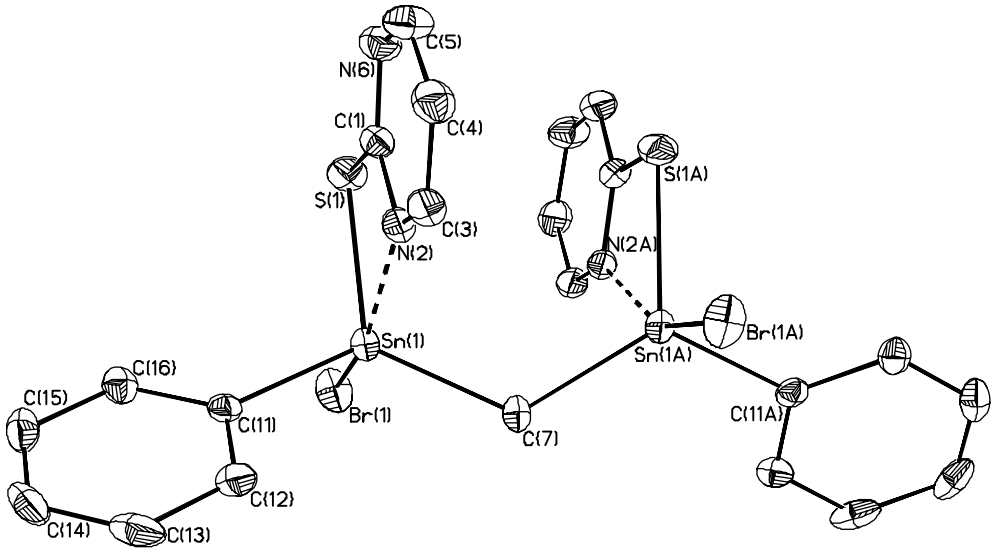


Fig. 3. General view (SHELXTL) of a molecule of **2** showing 30% probability displacement ellipsoids and the atom numbering scheme.

der Waals radii of the corresponding atoms. The molecule shows a *C*2/*c*-symmetry. The Sn(1) atom shows a distorted trigonal bipyramidal configuration (geometrical goodness $\Delta(\Sigma\vartheta)$ [35,36] 57.3°) with the S(1), C(7), and C(11) atoms occupying the equatorial and the Br(1) and N(2) atoms occupying the axial positions.

The Sn(1)–S(1) bond length is 2.4277(11) Å and is in agreement to those found in $[Ph_2(pmt)_2Sn]$ (Sn(1)–S(12) = 2.464(2) and Sn(1)–S(22) = 2.466(2) Å) [24], in $[Ph_3(pmt)Sn]$ (Sn–S(1) = 2.442(3) Å) [20] as well as to those found in $[Ph_2(Spy)_2Sn]$ (Sn(1)–S(1) = 2.485(1) and Sn(1)–S(2) = 2.476(2) Å) [19], in $[Cl_2(Spy)_2Sn]$ (Sn–S(1) = 2.467(3) and Sn–S(2) = 2.462(3) Å) [15] as well as in $[Ph(Spy)_3Sn\ 1.5CHCl_2]$ (Sn(1)–S(1) = 2.491(2), Sn(1)–S(2) = 2.562(2) and Sn(1)–S(3) = 2.576(2) Å) [22] where the ligands are coordinated to the tin atoms through their anionic forms but it is significant shorter to the corresponding value found in $[Cl_2Me_2(HSpy)_2Sn]$ (Sn–S(1) = 2.729(2) Å) [16] where pyridine-2-thione has been co-ordinated to the tin atom through its neutral form. The Sn(1)–N(2) bond length is 2.476(3) Å and it is in agreement to the bond distances found in $[Ph(Spy)_3Sn\ 1.5CHCl_2]$ (Sn(1)–N(11) = 2.425(5), Sn(1)–N(21) = 2.453(5) and Sn(1)–N(31) = 2.419(4) Å) [22] but it is longer to the corresponding value found in $[Cl_2(Spy)_2Sn]$ (Sn–N(1) = 2.271(9) and Sn–S(2) = 2.462(3) Å) [15] and it is shorter to those found in $[Ph_2(pmt)_2Sn]$ (Sn(1)–N(11) = 2.639(6) and Sn(1)–S(21) = 2.948(8) Å) [24] and in $[Me_2(Spy)_2Sn]$ (Sn–N = 2702(5) Å) [18]. The Sn(1)–C(7)–Sn(1A) bond angle amounts to 119.5(3)° which is bigger than the corresponding bond angle found in $[(Br_2PhSn)_2CH_2]$ (Sn(1)–C(1)–Sn(2) = 115.1(6)°) [37] and in $[(ClPh_2Sn)_2CH_2]$ (Sn(1)–C(1)–Sn(2) = 113.4(10)°) [38]. The C(1)–S(1)–Sn(1) bond angle is 87.37(13)° and similar to those found in $[Ph(Spy)_3Sn\ 1.5CHCl_2]$ C(11)–S(1)–Sn(1) = 85.1(2)°, C(21)–S(2)–Sn(1) = 85.2(2)° and C(31–S(3)–Sn(1) = 84.0(2)°) [22], in $[Me(Spy)_3Sn]$ (C(11)–S(1)–Sn(1) = 86.02(11)°, C(21)–S(2)–Sn(1) = 84.39(12)° and C(31)–S(3)–Sn(1) = 87.58(12)°) [22] and in $[Cl_2(Spy)_2Sn]$ (Sn–S(1)–C(1) = 81.9(4)°) [15] where the thione ligands are coordinated through their bidentate form but significantly shorter to those found in $[Cl_2Me_2(HSpy)_2Sn]$ (Sn–S(1)–C(2) = 105.1(3)°) [16] and in $[Ph_3(pmt)Sn]$ (Sn–S(1)–C(19) = 95.3(5)°) [20] where the thione ligands are bonded to the tin atom through their sulphur atoms.

4. Supporting information available

Supplementary data are available from CCDC, 12 Union Road, Cambridge CB2 1EZ, UK on request, quoting the deposition number CCDC 287122 for compound **1** and the deposition number CCDC 287121 for compound **2**.

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