Structural diversity of di and triorganotin complexes with 4-sulfanylbenzoic acid: Supramolecular structures involving intermolecular Sn···O, O–H···O or C–H···X (X = O or S) interactions

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Abstract

Twelve new organotin complexes with 4-sulfanylbenzoic acid of two types: $R_nSn[S(C_6H_4COOH)]_{4-n}$ (**I**) ($n = 3$: R = Me **1**, n-Bu **2**, Ph **3**; PhCH$_2$ **4**; $n = 2$: R = Me **5**; n-Bu **6**, Ph **7**, PhCH$_2$ **8**) and $R_3Sn(SC_6H_4COO)SnR_3 \cdot mEtOH$ (**II**) ($m = 0$: R = Me **9**, n-Bu **10**, PhCH$_2$ **12**; $m = 2$: R = Ph **11**), along with the 4,4′-bipy adduct of **9**, [Me$_3$Sn(SC$_6$H$_4$COO)SnMe$_3$]$_2$(4,4-bipy) **13**, have been synthesized. The coordination behavior of 4-sulfanylbenzoic acid is monodentate in **1–8** by thiol S atom but not carboxylic oxygen atom. While, in **9–13** it behaves as multidenate by both thiol S atom and carboxylic oxygen atoms. The supramolecular structures of **6**, **11** and **13** have been found to consist of 1D molecular chains built up by intermolecular O–H···O, C–H···O or C–H···S hydrogen bonds. The supramolecular aggregation of **7** is 2D network determined by two C–H···O hydrogen bonds. Extended intermolecular C–H···O interactions in the crystal lattice of **9** link the molecules into a 2D network.
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Keywords: 4-Sulfanylbenzoic acid; Organotin; Supramolecular structure

1. Introduction

Organotin complexes have attracted more and more attention in recent years, partly owing to their determinately or potentially pharmic value, where have been reported many before [1–4], and also for the versatile molecular structure and supramolecular architecture exhibited by these complexes [5,6]. It is the latter which forms the focus of this paper.

It is well known that the organotin carboxylates have versatile molecular structures both in solid and solution, such as monomers, dimers, tetramers, oligomeric ladders and hexameric durms, etc. [7]. It has also been demonstrated that other structural types are formed due to the presence of additional coordinating sites (S, N or O, etc.) along with a carboxylate moiety [5,6a,8].

In our previous work, we have reported several novel molecular structures of organotin complexes with 2-mercaptonicotinic acid [5a,9]. To continue our research, we have been attracted by another interesting ligand, 4-sulfanylbenzoic acid, recently. This ligand is interesting because of its potential multiple molecular models in organotin complexes. As exhibited in Scheme 1, at least seven possible molecular models are conceivable although nobody has investigated its actual molecular structures until now.

Although the synthesis and characterization of some transition metal complexes of 4-sulfanylbenzoic acid has
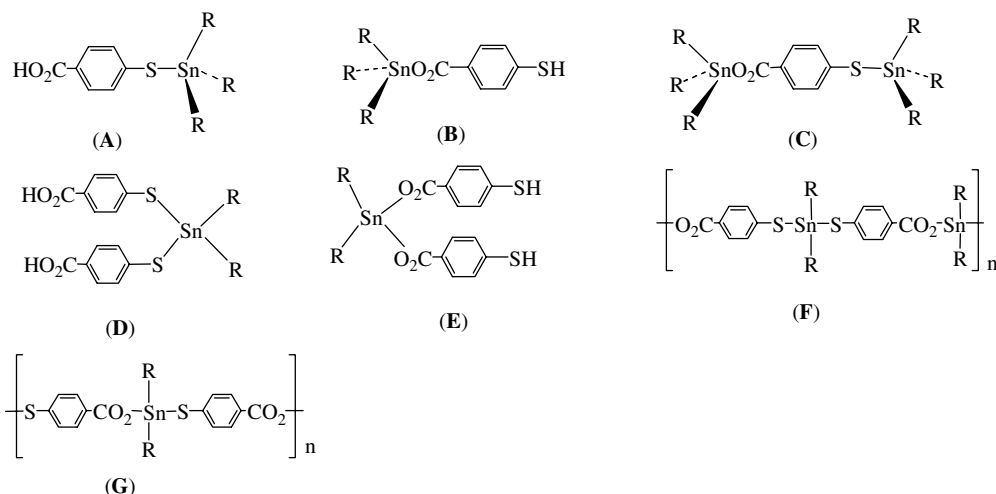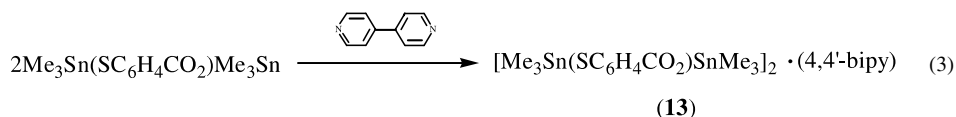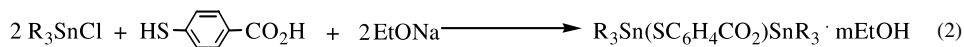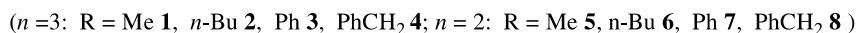
Scheme 1.

been carried out previously [10]. And to our knowledge, no organotin derivatives of 4-sulfanylbenzoic acid have been reported so far. Therefore, one of the aims of this work is to investigate and characterize the structures of new organotin derivatives of 4-sulfanylbenzoic acid.

Furthermore, supramolecular structure of organometallic complexes, especially aggregated through intermolecular soft hydrogen bonds [11], is of current interest and importance, which is mainly due to their intriguing, often complicated, architectures and topologies [12–15]. Ligands such as 4-sulfanylbenzoic acid present a number of opportunities for creating supramolecular arrangements via weak intermolecular metal–ligand interactions and/or H-bonding from Lewis base sites (O or S). Thus, the other aim of this work was to elucidate supramolecular structures of the organotin derivatives of 4-sulfanylbenzoic acid.

2. Results and discussion

2.1. Syntheses

Twelve new organotin complexes with 4-sulfanylbenzoic acid of two types: $R_nSn[S(C_6H_4CO_2H)]_{4-n}$ ($n = 3$:

$R = Me$ **1**, n-Bu **2**, Ph **3**; PhCH$_2$ **4**; $n = 2$: $R = Me$ **5**; n-Bu **6**, Ph **7**, PhCH$_2$ **8**) and $R_3Sn(SC_6H_4COO)SnR_3 \cdot$ mEtOH (**II**) ($m = 0$: $R = Me$ **9**, n-Bu **10**, PhCH$_2$ **12**; $m = 2$: $R = Ph$ **11**), have been synthesized by reaction of the 4-sulfanylbenzoic acid, sodium ethoxide and the corresponding organotin chlorides (Eqs. (1) and (2)). Further reaction of **9** with excess 4,4'-bipy in diethyl ether results in the formation of a tetranuclear adduct, $[Me_3Sn(SC_6H_4CO_2)SnMe_3]_2 \cdot (4,4'-bipy)$ (**13**) (Eq. (3)). The syntheses procedure is given in Scheme 2.

2.2. IR Spectra

The explicit feature in the IR spectra of the 13 complexes **1**–**13** is the absence of the band in the region 2550–2420 cm^{-1}, which appears in the free ligand as the -SH stretching vibration, thus indicating metal–ligand bond formation through this site. In the far-IR spectra, the absorption about 305–316 cm^{-1} region for all complexes **1**–**13**, which is absent in the spectrum of the ligand, is assigned to the Sn–S stretching mode of the vibration and all the values are located within the range for Sn–S vibration observed in common organotin derivatives of thiolate (300–400 cm^{-1}) [16,17].

$$R_nSnCl_{4-n} + (4\text{-}n)\,HS\!-\!\!\bigcirc\!\!-CO_2H + (4\text{-}n)\,EtONa \longrightarrow R_nSn[S(C_6H_4CO_2H)]_{4-n} \quad (1)$$

($n = 3$: $R = Me$ **1**, n-Bu **2**, Ph **3**, PhCH$_2$ **4**; $n = 2$: $R = Me$ **5**, n-Bu **6**, Ph **7**, PhCH$_2$ **8**)

$$2\,R_3SnCl + HS\!-\!\!\bigcirc\!\!-CO_2H + 2\,EtONa \longrightarrow R_3Sn(SC_6H_4CO_2)SnR_3 \cdot mEtOH \quad (2)$$

(m = 0: $R = Me$ **9**, n-Bu **10**, PhCH$_2$ **12**; m = 2: $R = Ph$ **11**,)

$$2Me_3Sn(SC_6H_4CO_2)Me_3Sn \xrightarrow{\quad\bigcirc\!-\!\bigcirc\quad} [Me_3Sn(SC_6H_4CO_2)SnMe_3]_2 \cdot (4,4'-bipy) \quad (3)$$

(**13**)

Scheme 2.

Moreover, the broad absorption at 2900–3100 cm^{-1} is present in complexes **1–8**, which is assigned to –CO$_2$H groups. While it is absent in complexes **9–13**, indicating the deprotonation and coordination of these carboxylate groups.

Furthermore, in organotin carboxylate complexes, the IR spectroscopy can provide useful information concerning the coordinate formation of the carboxylate [18]. When the carboxylate group coordinates the tin atom in a monodentate manner (Scheme 3 mode I), the difference between the wavenumbers of the asymmetric and symmetric carboxylate stretching bonds, $\Delta v[v(COO)_{as} - v(COO)_s]$, is larger than that observed for ionic complexes. When the ligand chelates (mode II), Δv is considerably smaller than that for ionic complexes [19]. The characteristic wavenumber difference, Δv, for mode III is larger than that for chelated ions and nearly the same as observed for ionic complexes.

Based on the above mention, it was possible to distinguish the coordination mode of the –CO$_2$ group. The Δv values for complexes **9** and **10** (200 and 190 cm^{-1}) are near to that in the sodium salt of the ligand (205 cm^{-1}), which suggest that the coordinate formation of these carboxylate groups is mode III. While the Δv values for complexes **11**, **12** and **13** (157, 150 and 165 cm^{-1}) are smaller than that in the sodium salt of the ligand, which suggest that the coordinate formation of these carboxylate groups is mode II. All the Δv values of complexes **9–13**, compared with those for the corresponding sodium salts, reveal that the –CO$_2$ group of 4-sulfanylbenzoic acid functions as bidentate under the conditions employed [20,21]. This information is consistent with the analysis of the related X-ray crystallography.

2.3. ^{1}H NMR spectra

^{1}H NMR data showed that the signal of the –SH proton (1.65 ppm) in the spectrum of the ligand is absent in all of the complexes, indicating the removal of the –SH proton and the formation of Sn–S bonds. Moreover, the sharp resonance appears at about 10.5 ppm in the spectra of **1–8**, which is assigned to CO$_2$H. While it is absent in **9–13**, indicating the replacement of the carboxylic acid proton by R$_n$Sn ($n = 2$ or 3) moiety. Signals for the other groups appear at the same position as in the ligand. All the information accords well with what the IR data have revealed.
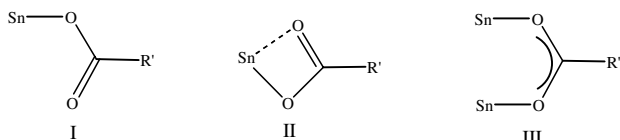
Scheme 3.

2.4. Structural chemistry

2.4.1. Ph$_3$Sn[S(C$_6$H$_4$CO$_2$H)] (3)

The molecular structure of complex **3** is shown in Fig. 1, selected bond lengths and angles are given in Table 1. The central tin atom of complex **3** forms four primary bonds: three to the phenyl groups and one to the sulfur atom (model **A**). Thus, complex **3** displays a distorted tetrahedral coordination sphere with six angles ranging from 99.08(8) to 119.44(12)°. The Sn–C bond lengths [2.123(3)–2.135(3) Å] are in the range of that found in other triphenyltin thiolate (2.115–2.138 Å) [22]. The Sn–S bond length [2.4272(13) Å] is a little longer that has reported in methylthio-triphenyl-tin (2.390 Å) [22] and approaches the sum of the covalent radii of tin and sulfur (2.42 Å) [23].

2.4.2. (n-Bu)$_2$Sn[S(C$_6$H$_4$CO$_2$H)]$_2$ (6) and (Ph)$_2$Sn[S(C$_6$H$_4$CO$_2$H)]$_2$ (7)

The molecular structure of complexes **6** and **7** are shown in Figs. 2 and 3, selected bond lengths and angles are given in Tables 2 and 3. The geometries at central Sn atoms of both complexes **6** and **7** are also distorted tetrahedron but formed by two S atoms from 4-sulfanylbenzoic acid and two C atoms form butyl groups or phenyl groups (model **D**). The C–Sn–C angle [113.8(6)° for **6**, 116.44(19)° for **7**] is nearly to that found in similar diorganotin dithiolate (116.43–126.72°) [24–26]. The S–Sn–S

Fig. 1. Molecular structure of the complex **3** with thermal ellipsoids at 30% probability level.

Table 1
Selected bond lengths (Å) and angles (°) for **3**

Sn1–C8	2.133(3)	Sn1–C14	2.135(3)
Sn1–C20	2.123(3)	Sn1–S1	2.4272(13)
C200–Sn1–C8	109.37(12)	C20–Sn1–C14	110.11(12)
C8–Sn1–C14	119.44(12)	C20–Sn1–S1	109.63(9)
C8–Sn1–S1	108.52(9)	C14–Sn1–S1	99.08(8)

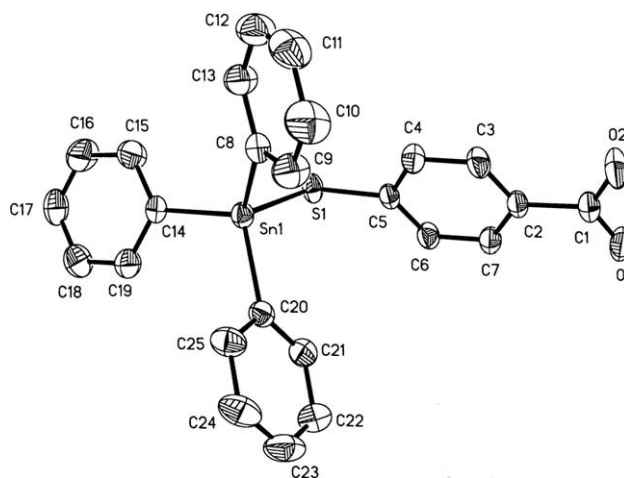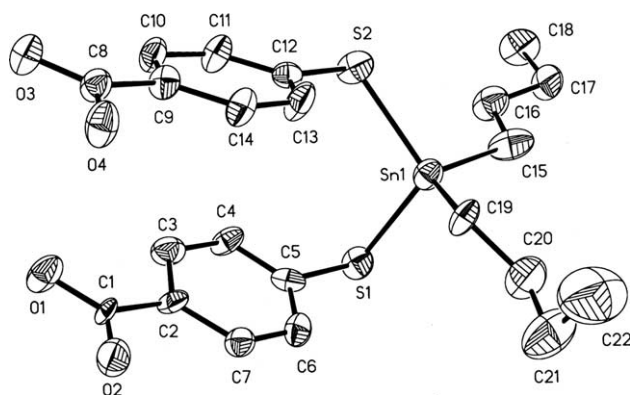Fig. 2. Molecular structure of the complex **6** with thermal ellipsoids at 30% probability level.
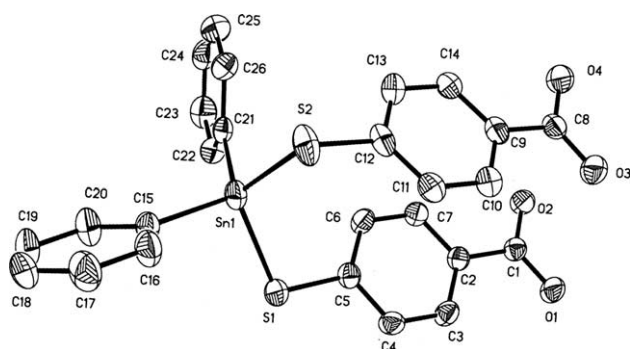

Fig. 3. Molecular structure of the complex **7** with thermal ellipsoids at 30% probability level.

Table 2
Selected bond lengths (Å) and angles (°) for **6**

Sn1–C15	2.099(16)	Sn1–C19	2.189(13)
Sn1–S1	2.429(7)	Sn1–S2	2.414(8)
C15–Sn1–C19	113.8(6)	C15–Sn1–S2	102.7(4)
C19–Sn1–S2	114.3(3)	C15–Sn1–S1	110.7(4)
C19–Sn1–S1	108.2(4)	S2–Sn1–S1	106.91(17)

Table 3
Selected bond lengths (Å) and angles (°) for **7**

Sn1–C21	2.121(5)	Sn1–C15	2.127(5)
Sn1–S1	2.4108(16)	Sn1–S2	2.4104(16)
C21–Sn1–C15	116.44(19)	C21–Sn1–S1	110.89(15)
C15–Sn1–S1	102.51(15)	C21–Sn1–S2	109.60(15)
C15–Sn1–S2	103.66(15)	S1–Sn1–S2	113.55(6)

angle [113.55(6)° for **6** and 106.91(17)° for **7**] is close to that found in bis(benzenethiolato)-diphenyl-tin (110.79°) [27], and markedly larger than that found in chelate diorganotin dithiolates (90.54–92.48°) [25,26]. The related C–Sn–S angles [102.7(4)–114.3(3)° for **6**, 102.51(15)–110.89(15)° for **7**] are nearly to the theoretical tetrahedral angle. Both the Sn–C [2.099(16) and 2.189(13) Å for **6**, 2.121(5) and 2.127(5) Å for **7**] and

the Sn–S bond lengths [2.414(8) and 2.429(7) Å for **6**, 2.4104(16) and 2.4108(16) Å for **7**] are excellent agreement with other previously known organotin thiolates in the literatures [24–27].

2.4.3. $Me_3Sn(SC_6H_4CO_2)Me_3Sn$ (**9**) and $Ph_3Sn(SC_6H_4CO_2)Ph_3Sn \cdot 2EtOH$ (**11**)

The molecular structure of complexes **9** and **11** are shown in Figs. 4 and 5, selected bond lengths and angles are given in Tables 4 and 5. There are two types of independent Sn atoms in both complexes **9** and **11**: one is the four-coordinated Sn atom connected by thiol S atom,
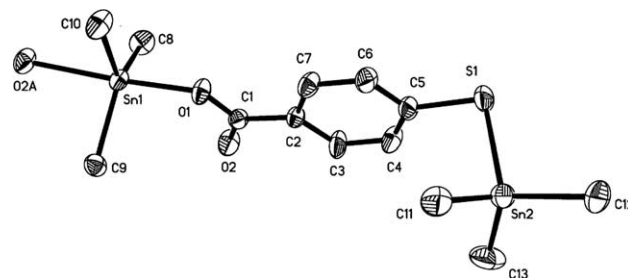

Fig. 4. Molecular structure of the complex **9** with thermal ellipsoids at 30% probability level.
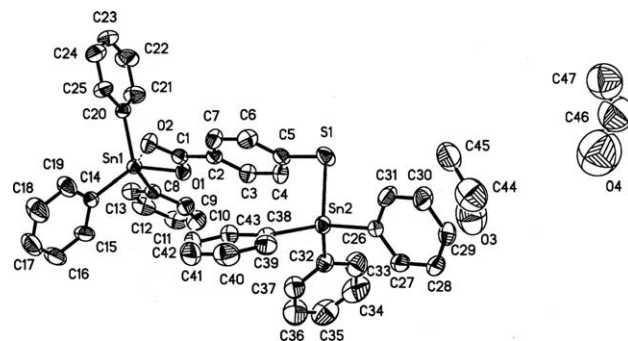

Fig. 5. Molecular structure of the complex **11** with thermal ellipsoids at 30% probability level.

Table 4
Selected bond lengths (Å) and angles (°) of **9**

Sn1–C10	2.110(7)	Sn1–C8	2.121(7)
Sn1–C9	2.131(7)	Sn1–O1	2.140(4)
Sn1–O2A	2.504(5)	Sn2–C13	2.126(9)
Sn2–C12	2.137(8)	Sn2–C11	2.139(10)
Sn2–S1	2.451(2)	O1–C1	1.278(8)
O2–C1	1.232(8)		
C10–Sn1–C8	116.0(4)	C10–Sn1–C9	117.1(3)
C8–Sn1–C9	125.5(3)	C10–Sn1–O1	88.7(2)
C8–Sn1–O1	97.4(3)	C9–Sn1–O1	95.2(2)
C10–Sn1–O2A	87.1(2)	C9–Sn1–O2A	87.6(2)
O1–Sn1–O2A	175.60(17)	C13–Sn2–C12	112.2(5)
C13–Sn2–C11	113.5(4)	C12–Sn2–C11	112.2(4)
C13–Sn2–S1	108.1(3)	C12–Sn2–S1	106.9(3)
C11–Sn2–S1	103.3(3)		

Table 5
Selected bond lengths (Å) and angles (°) for **11**

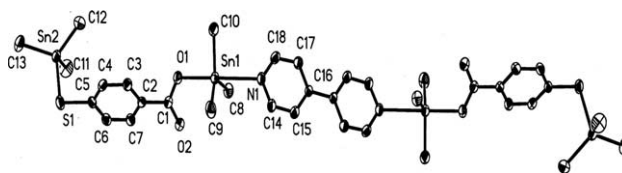|			
Sn1–C8	2.131(5)	Sn1–C14	2.118(5)
Sn1–C20	2.118(5)	Sn1–O1	2.055(3)
Sn1–O2	2.737(4)	Sn2–C26	2.117(5)
Sn2–C32	2.128(6)	Sn2–C38	2.135(5)
Sn2–S1	2.4227(17)	O1–C1	1.312(6)
O2–C1	1.227(6)		
O1–Sn1–C20	108.98(16)	O1–Sn1–C14	111.77(17)
C20–Sn1–C14	117.2(2)	O1–Sn1–C8	96.13(18)
C20–Sn1–C8	111.3(2)	C14-Sn1–C8	109.5(2)
O1–Sn1–O2	52.55(13)	C20–Sn1–O2	83.26(16)
C14-Sn1–O2	85.67(17)	C8–Sn1–O2	148.68(18)
C26–Sn2–C32	113.4(2)	C26–Sn2–C38	113.4(2)
C26–Sn2–S1	102.05(15)	C32–Sn2–S1	109.17(16)
C38–Sn2–S1	109.76(14)		

Fig. 6. Molecular structure of the complex **13** with thermal ellipsoids at 30% probability level.

Table 6
Selected bond lengths (Å) and angles (°) for **13**

Sn1–C10	2.117(5)	Sn1–C9	2.123(5)
Sn1–C8	2.109(5)	Sn1–O1	2.137(3)
Sn1–N1	2.631(4)	Sn1–O2	3.151(4)
Sn2–C13	2.124(5)	Sn2–C12	2.127(6)
Sn2–C11	2.114(6)	Sn(2)–S(1)	2.4338(15)
C10–Sn1–C9	116.1(2)	C10–Sn1–C8	117.1(2)
C9–Sn1–C8	124.6(2)	C10–Sn1–O1	90.45(17)
C9–Sn1–O1	94.33(19)	C8–Sn1–O1	99.64(18)
C10–Sn1–N1	90.03(17)	C9–Sn1–N1	86.22(18)
C8–Sn1–N1	79.38(17)	O1–Sn1–N1	179.02(13)
C9–Sn1–O2	82.46(19)	C8–Sn1–O2	71.61(16)
C10–Sn1–O2	134.30(16)	O1–Sn1–O2	44.90(11)
N1–Sn1–O2	134.46(11)	C13–Sn2–C12	112.2(3)
C13–Sn2–C11	113.8(3)	C12–Sn2–C11	113.1(3)
C13–Sn2–S1	104.4(2)	C12–Sn2–S1	106.10(18)
C11–Sn2–S1	106.3(2)		

the other is the five-coordinate Sn atom connected by carboxyl O atoms (model **C**). The geometries of Sn2 atoms of complexes **9** and **11** are both distorted tetrahedral, and these bond lengths and bond angles are similar to those found in complex **3**. Moreover, the geometry at Sn1 atom of complex **9** is distorted trigonal bipyramidal with three methyl groups occupying equatorial plane and two O atoms in axial sites [O1–Sn1–O2A, 175.60(17)°]. The geometry at Sn1 atom of complex **11** is also distorted trigonal bipyramidal but with two O atoms of carboxyl groups and one C atom of phenyl group occupying equatorial plane and two C atoms of phenyl groups in axial sites [C20–Sn1–C14, 117.2(2)°]. Therefore, the geometries of Sn1 atoms of complexes **9** and **11** are not identical: *trans*-O_2SnC_3 type [28] for **9** and *cis*-O_2SnC_3 type [29] for **11**. The Sn–C distances [2.110(7)–2.131(7) Å for **9**, 2.118(5)–2.131(5) Å for **11**] are consistent with those reported in other triorganotin carboxylates [28,29]. The Sn1–O1 distance [2.140(4) Å for **9**, 2.055(3) Å for **11**] is a little shorter than that reported in [Me$_3$Sn(O$_2$CC$_4$H$_3$S)] (2.149 Å) [30] and [Ph$_3$Sn(O$_2$CC$_6$H$_5$)] (2.073 Å) [31] and approaches the sum of the covalent radii of Sn and O (2.13 Å) [32]. The Sn1–O2 distance [2.504(5) Å for **9**, 2.737(4) Å for **11**] is close to the distance between coordinated O atom and central Sn atom in other organotin complexes [30,31], but large shorter than the sum of the van der waals radii of Sn and O, 3.68 Å [32].

Besides, it should be noted that the component Ph$_3$Sn(SC$_6$H$_4$CO$_2$)Ph$_3$Sn crystallizes with two solvent molecules of EtOH, but it has not been found the obvious interactions between the Ph$_3$Sn(SC$_6$H$_4$CO$_2$)Ph$_3$Sn moiety and the solvent molecules.

2.4.4. [Me₃Sn(SC₆H₄COO)Me₃Sn]₂·(4,4′-bipy) (13)

The molecular structure of complex **13** is shown in Fig. 6, selected bond lengths and angles are given in Table 6. The asymmetric unit of **13** was found to consist of one-half of the tetranuclear molecule, the remainder being generated by an inversion center at the midpoint

of the 4,4′-bipy ligand. The structure contains two Me$_3$Sn(SC$_6$H$_4$CO$_2$)SnMe$_3$ units associated through a bridging 4,4′-bipy moiety. The coordination environment around tin is *trans*-NOSnC$_3$ with the axial positions occupied by the carboxyl O atom of 4-sulfanylbenzoic acid and the ring N atom of the neutral bipyridyl donor. The O–Sn–N [179.02(13)°] and C–Sn–C angles [116.1(2)–124.6(2)°] reflect this coordination geometry. The Sn–O bond length [2.137(3) Å] is almost near to that of **9** [2.140(4) Å] and at the longer end of the range of Sn–O distances measured for triorganotin carboxylate [28–31]. The Sn–N bond length [2.631(4) Å], closely to the Sn–N distance in [Me$_3$Sn(PhN$_2$C$_2$S$_3$)]$_2$· (4,4′-bipy) [2.613(2) Å] [14], is longer than those found in organotin tetrazoles [2.27–2.43 Å] [33] or the cationic polymer [Me$_3$Sn(4,4′-bipy)]$_n^{n+}$ [2.411, 2.420 Å] [34]. Furthermore, it is noteworthy that a weak intramolecular Sn···O interaction is recognized between the Sn1 and the O2 derived from the monodentate carboxyl group. The Sn1···O2 distance [3.151(4) Å], which is longer than that of **11** [2.737(4) Å], is considerably less than the sum of the van der waals radii (3.68 Å) [32]. Thus, if the weak Sn1···O2 interaction is considered, the geometry of Sn1 is best described as distorted octahedron.

2.5. Supramolecular structures

As shown in Fig. 7, a pair of intermolecular O–H···O hydrogen bonds are recognized, which associate the
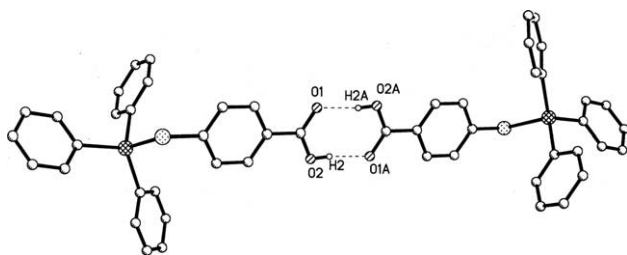
Fig. 7. Dimer structure of the complex **3**, showing intermolecular O–H···O hydrogen bonding.

discrete molecule into a dimer. This is similar to that found in $Au(Et_3P)(4\text{-}SC_6H_4CO_2H)$ [10]. The O···O and H···O distance and the O–H···O angle are 2.644 and 1.829 Å and 171.99°, respectively.

As shown in Fig. 8(a), through the dimerization of the two molecules of complex **6**, the two benzoic acid units become stacked on top of each other, however, the phenylene rings are not coplanar and have a distance between their centers of 3.9575 Å, which is a little shorter than that found in $Au(Ph_3P)(4\text{-}SC_6H_4CO_2H)$ (4.041 Å) [10]. The carboxylic acid end groups are able to engage in parallel paired hydrogen bonding with another molecular unit. The distance between the stacked hydrogen-bonded systems is 3.8705 Å, and thus a little shorter than the distance between the stacked phenylene units. In total, the complex **6** forms dimer through a large elongated macrocycle comprising two tin atoms and two pairs of 4-sulfanylbenzoic acid groups. These

dimers of compelx **6** are further linked by intermolecular C–H···O hydrogen bonds. As shown in Fig. 8(b), the C18 atom acts as a hydrogen-bond donor via H18C atom to carboxyl O3B atom, and the C18B atom also acts as a hydrogen-bond donor via H18B atom to carboxyl O3, so forming a centrosymmetric macrocycle with a head-to-tail arrangement [35]. The C···O and H···O distances and the C–H···O angle are 3.608 and 2.677 Å and 163.57°, which is consistent with that reported in 2-nitrophenoxyacetanilide (C···O = 3.5006(15) Å, H·O = 2.55 Å, C–H···O = 162°) [36]. Despite these weak C–H···O interactions often being neglected, they clearly govern these self-assembly of the molecules into 1D chain (Fig. 8(b)).

The two molecules of complex **7** also form a macrocyclic dimer (Fig. 9(a)), which is similar to that found in complex **6**. The dimers of complex **7** are linked by two intermolecular C–H···O hydrogen bonds, one a little weaker than the other. The stronger one (C25A···O1C = 3.276 Å, H25A···O1C = 2.568 Å, C25A–H25A···O1C = 133.30°) forms a chain of rings (Fig. 9(b)), which is similar to that found in complex **6**. Besides, these dimers of complex **7** are linked by the longer one [C22A···O4C = 3.423 Å, H22A···O4C = 2.636 Å, C22A–H22A···O4C = 142.77°], also forming a chain of rings [Fig. 9(c)]. Therefore, a 2D network (Fig. 9(d)) has been found in complex **7**.

The intermolecular C=O → Sn coordination in **9** leads to infinite zigzag chains containing the metal centers and carboxyl groups (Fig. 10(a)), which is similar to
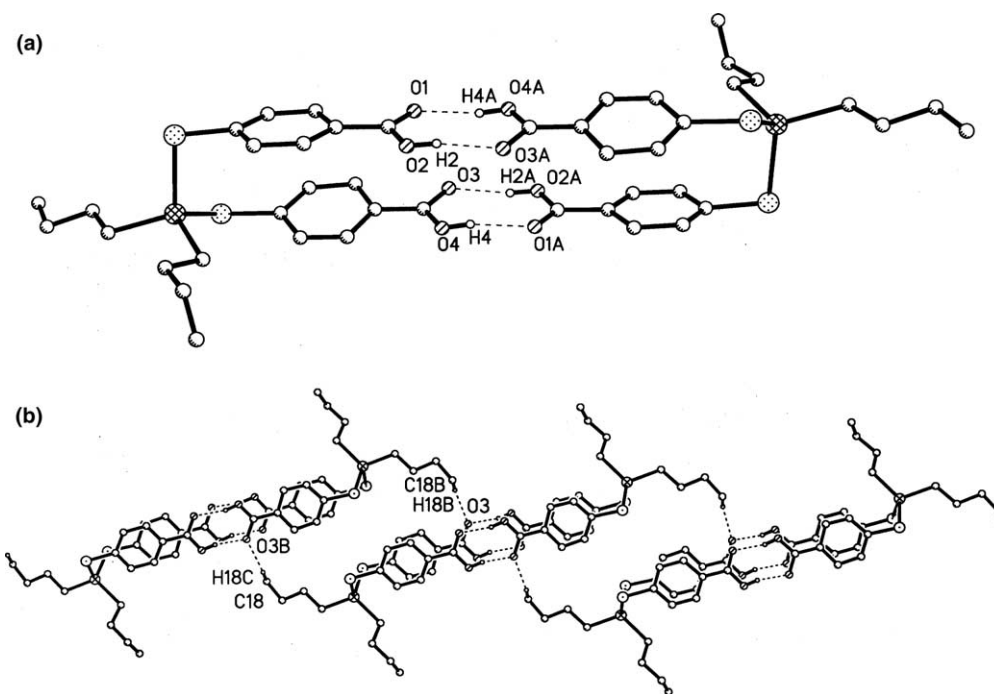

Fig. 8. The supramolecular structure of the complex **6**, showing (a) macrocyclic dimer and (b) 1D chain of rings connected by intermolecular O–H···O and C–H···O hydrogen bonding.

Fig. 10. Supramolecular structure of complex **9**, showing (a) polymer propagation via O→Sn coordination and (b) additional inter-chain C–H···O hydrogen bonding.
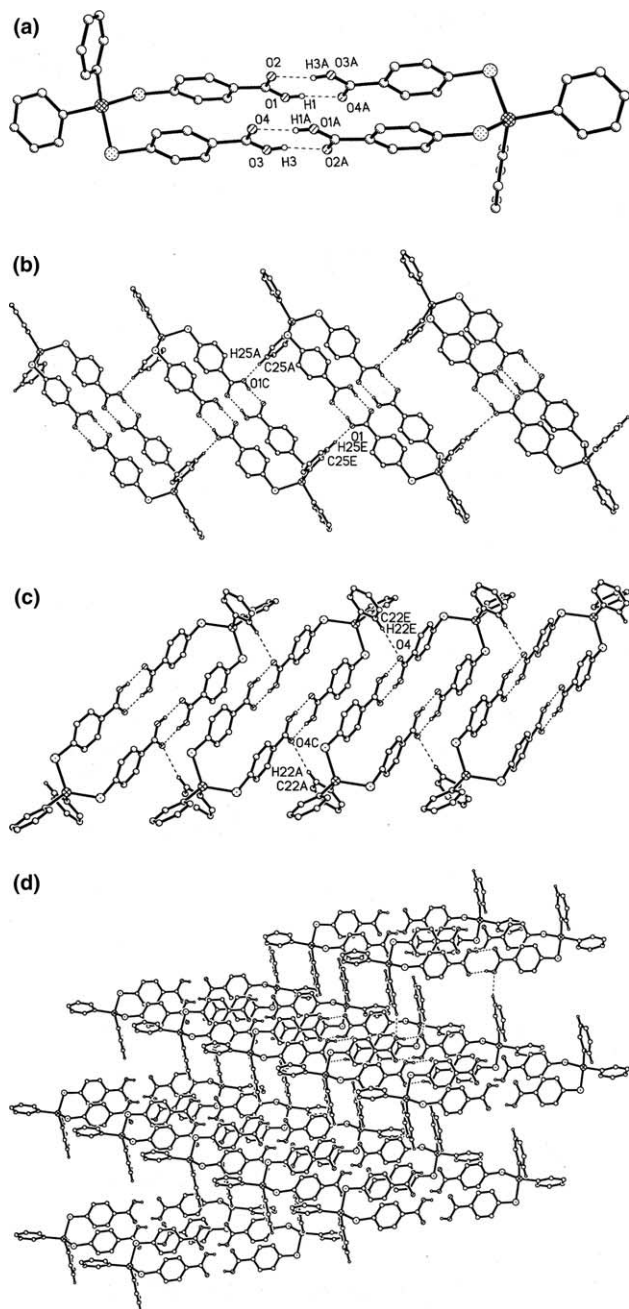
Fig. 9. Supramolecular structure of complex **7**, showing (a) macrocyclic dimer, (b) 1D chain of rings along *a* axis, (c) 1D chain of rings along *b* axis and (d) a 2D network in *ab* plane.

another organotin that has been reported [28]. Furthermore, the O atoms of the carboxyl groups and the C atoms of methyl groups interconnect at 3.499 Å, so forming intermolecular C–H···O hydrogen bonds (C11···O1 = 3.499 Å, H11···O1 = 2.706 Å, C11–H11···O1 = 140.24°). This structural feature leads to an expansion of the supramolecular structure of **9** to a 2D network (Fig. 10(b)).

The supramolecular structure of **11** is dominated by one-dimensional helical polymer (Fig. 11) along the *b*
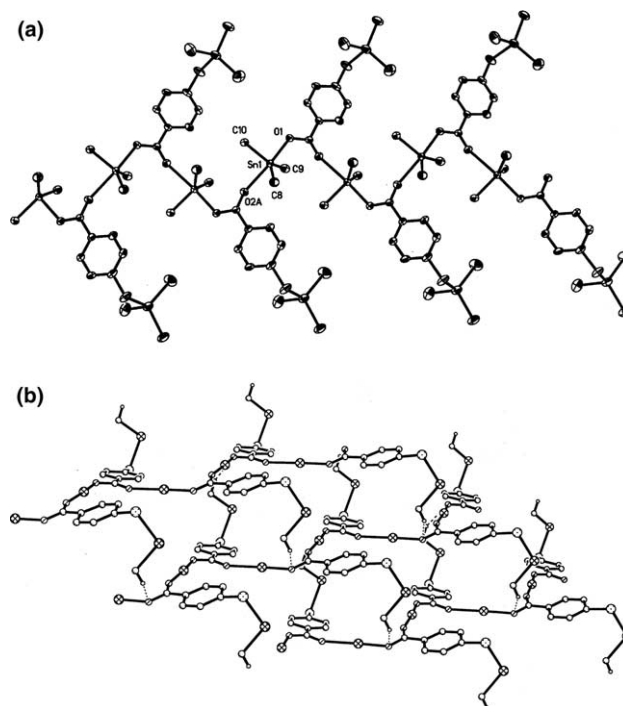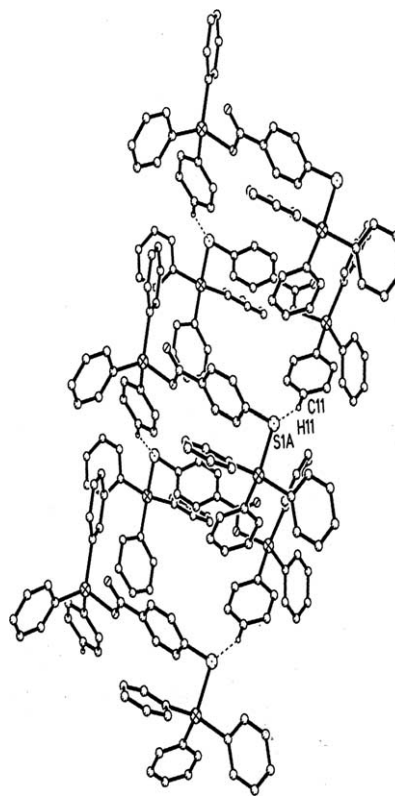

Fig. 11. Supramolecular structure of complex **11**, showing a hical chain along *c* connected by intermolecular C–H···S hydrogen bonds.
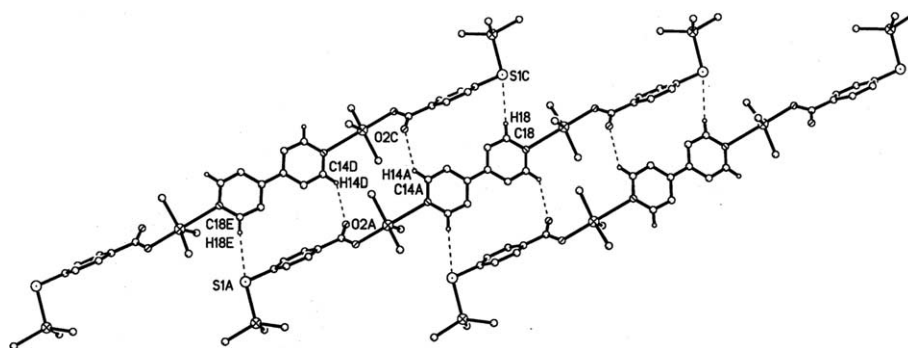
Fig. 12. Supramolecular structure of complex **13**, showing 1D chain of rings connected by intermolecular C–H$\cdots$O and C–H$\cdots$S hydrogen bonding.

aixs linked by intermolecular hydrogen bonds C–H$\cdots$S (C11A$\cdots$S1 = 3.821 Å, H11A$\cdots$S1 = 2.957 Å, C11A–H11A$\cdots$S1 = 155.18°). The 'pitch' of the helix (9.991 Å) is large shorter than that found in 4-[2-(triethylstannyl)tetrazol-5-yl] pyridine (40 Å) [37], which is possibly due to the different molecular conformation and bonding mode between the two complexes.

The salient feature of the supramolecular structure of complex **13** is that of a 1D polymer linked by intermolecular C–H$\cdots$O and C–H$\cdots$S hydrogen bonds. As shown in Fig. 12, a 14-membered ring is formed by two intermolecular C–H$\cdots$O (C14A$\cdots$O2C = 3.195 Å, H14A$\cdots$O2C = 2.469 Å, C14A–H14A$\cdots$O2C = 134.95°) hydrogen bonds with a head-to-tail arrangement. Besides, the adjacent intermolecular C–H$\cdots$S (C18$\cdots$S1C = 3.755 Å, H18$\cdots$S1C = 2.959 Å, C18–H18$\cdots$S1C = 144.62°) hydrogen bonds also form similar 32-membered ring. Thus, intermolecular quadruplex hydrongen bonds have been found in complex **13**, which help the construction of concentric annular structure. To our knowledge, this similar structure has not been reported so far.

3. Experimental

3.1. Materials and measurements

Trimethyltin chloride, tri-*n*-butyltin chloride, triphenyltin chloride, dimethyltin dichloride, di-*n*-butyltin dichloride, diphenyltin dichloride and 4-sulfanylbenzoic acid were commercially available, and they were used without further purification. Tribenzyltin chloride and dibenzyltin dichloride were prepared by a standard method reported in the literature [38]. The melting points were obtained with Kofler micro melting point apparatus and are uncorrected. Infrared spectra were recorded on a Nicolet-460 spectrophotometer using KBr discs and sodium chloride optics. ^{1}H NMR spectra were obtained on a JEOL-FX-90Q spectrometer, chemical shifts were given in ppm relative to Me$_4$Si in CDCl$_3$ solvent. Elemental analyses were performed with a PE-2400II apparatus.

3.2. Syntheses

3.2.1. Me$_3$Sn[S(C$_6$H$_4$CO$_2$H)] (1)

The reaction was carried out under nitrogen atmosphere. The 4-sulfanylbenzoic acid (0.154 g, 1 mmol) was added to the solution of benzene 20 ml with sodium ethoxide (0.068 g, 1 mmol), and the mixture was stirred for 10 min, then added Me$_3$SnCl (0.199 g, 1 mmol) to the mixture, continuing the reaction for 12 h at 40 °C. After cooling down to the room temperature, the solution was filtered. The solvent of the filtrate was gradually removed by evaporation under vacuum until solid product was obtained. The solid was then recrystallized from ether. Colorless crystal was formed. Yield: 85%. m.p. 98–100 °C. *Anal*. Calc. for C$_{10}$H$_{14}$O$_2$SSn: C, 37.89; H, 4.45. Found: C, 37.80; H, 4.38%. ^{1}H NMR (CDCl$_3$, ppm): δ 10.55 (s, 1H) 7.23–7.38 (m, 4H), 0.75 (s, 9H). IR (KBr, cm^{-1}): 2980, 1725, 545, 520, 312.

3.2.2. (n-Bu)$_3$Sn[S(C$_6$H$_4$CO$_2$H)] (2)

The synthesis procedure was the same as **1**. 4-Sulfanylbenzoic acid (0.154 g, 1 mmol), sodium ethoxide (0.068 g, 1 mmol) and (*n*-Bu)$_3$SnCl (0.326 g, 1 mmol), reaction time 12 h, temperature 40 °C. Recrystallized from ether–petroleum. Colorless crystal was formed. Yield: 78%. m.p. 70–72 °C. *Anal*. Calc. for C$_{19}$H$_{32}$O$_2$SSn: C, 51.49; H, 7.28. Found: C, 51.50; H, 7.32%. ^{1}H NMR (CDCl$_3$, ppm): δ 10.48 (s, 1H) 7.25–7.38 (m, 4H), 0.84–1.71 (m, 27H). IR (KBr, cm^{-1}): 3100, 1720, 548, 526, 308.

3.2.3. Ph$_3$Sn[S(C$_6$H$_4$CO$_2$H)] (3)

The synthesis procedure was the same as **1**. 4-Sulfanylbenzoic acid (0.154 g, 1 mmol), sodium ethoxide (0.068 g, 1 mmol) and Ph$_3$SnCl (0.385 g, 1 mmol), reaction time 12 h, temperature 40 °C. Recrystallized from hexane-dichloromethane. Colorless crystal was formed. Yield: 80%. m.p. 178–180 °C. *Anal*. Calc. for C$_{25}$H$_{20}$O$_2$SSn: C, 59.67; H, 4.01. Found: C, 59.71; H, 3.98%. ^{1}H NMR (CDCl$_3$, ppm): δ 10.58 (s, 1H) 7.18–7.37 (m, 4H), 7.46–7.79 (m, 15H). IR (KBr, cm^{-1}): 3000, 1718, 546, 519, 310.

3.2.4. (PhCH₂)₃Sn[S(C₆H₄CO₂H)] (4)

The synthesis procedure was the same as **1**. 4-Sulfanylbenzoic acid (0.154 g, 1 mmol), sodium ethoxide (0.068 g, 1 mmol) and (PhCH₂)₃SnCl (0.427 g, 1 mmol), reaction time 12 h, temperature 40 °C. Recrystallized from dichloromethane. Colorless crystal was formed. Yield: 75%. m.p. 134–136 °C. *Anal.* Calc. for C₂₈H₂₆O₂SSn: C, 61.67; H, 4.81. Found: C, 61.77; H, 4.86%. ¹H NMR (CDCl₃, ppm): δ 10.52 (s, 1H) 7.30 (m, 4H), 6.88–7.15 (m, 15H), 2.78 (s, 6H). IR (KBr, cm⁻¹): 3015, 1722, 538, 513, 307.

3.2.5. Me₂Sn[S(C₆H₄CO₂H)]₂ (5)

The synthesis procedure was the same as **1**. 4-Sulfanylbenzoic acid (0.308 g, 2 mmol), sodium ethoxide (0.136 g, 2 mmol) and Me₂SnCl₂ (0.220 g, 1 mmol), reaction time 12 h, temperature 40 °C. Recrystallized from ether–petroleum. Colorless crystal was formed. Yield: 88%. m.p. 124–126 °C. *Anal.* Calc. for C₁₆H₁₈O₄S₂Sn: C, 34.74; H, 3.28; N, 20.26. Found: C, 34.84; H, 3.23; N, 20.07%. ¹H NMR (CDCl₃, ppm): δ 10.54 (s, 2H) 7.26–7.40 (m, 8H), 1.42 (s, 6H). IR (KBr, cm⁻¹): 2900, 1710, 543, 517, 305.

3.2.6. (n-Bu)₂Sn[S(C₆H₄CO₂H)]₂ (6)

The synthesis procedure was the same as **1**. 4-Sulfanylbenzoic acid (0.308 g, 2 mmol), sodium ethoxide (0.136 g, 2 mmol) and (n-Bu)₂SnCl₂ (0.304 g, 1 mmol), reaction time 12 h, temperature 40 °C. Recrystallized from ether–petroleum. Colorless crystal was formed. Yield: 83%. m.p. 128–130 °C. *Anal.* Calc. for C₂₂H₂₈O₄S₂Sn: C, 49.00; H, 5.23. Found: C, 48.95; H, 5.25%. ¹H NMR (CDCl₃, ppm): δ 10.46 (s, 2H) 7.20–7.37 (m, 8H), 0.86–2.12 (m, 18H). IR (KBr, cm⁻¹): 2955, 1715, 550, 525, 315.

3.2.7. Ph₂Sn[S(C₆H₄CO₂H)]₂ (7)

The synthesis procedure was the same as **1**. 4-Sulfanylbenzoic acid (0.308 g, 2 mmol), sodium ethoxide (0.136 g, 2 mmol) and Ph₂SnCl₂ (0.344 g, 1 mmol), reaction time 12 h, temperature 45 °C. Recrystallized from ether–petroleum. Colorless crystal was formed. Yield: 75%. m.p. 168–170 °C. *Anal.* Calc. for C₂₆H₂₀O₄S₂Sn: C, 53.91; H, 3.48. Found: C, 53.88; H, 3.54%. ¹H NMR (CDCl₃, ppm): δ 10.57 (s, 2H) 7.20–7.33 (m, 8H), 7.46–7.79 (m, 10H). IR (KBr, cm⁻¹): 3005, 1724, 548, 529, 309.

3.2.8. (PhCH₂)₂Sn[S(C₆H₄CO₂H)]₂ (8)

The synthesis procedure was the same as **1**. 4-Sulfanylbenzoic acid (0.308 g, 2 mmol), sodium ethoxide (0.136 g, 2 mmol) and (PhCH₂)₂SnCl₂ (0.372 g, 1 mmol), reaction time 12 h, temperature 40 °C. Recrystallized from ether–petroleum. Colorless crystal was formed. Yield: 80%. m.p. 102–104 °C. *Anal.* Calc. for C₂₈H₂₄O₄S₂Sn: C, 55.37; H, 3.98. Found: C, 55.35; H, 3.99%. ¹H NMR (CDCl₃, ppm): δ 10.52 (s, 2H) 7.17–7.42 (m, 8H), 6.82–7.05 (m, 10H), 3.52 (s, 4H). IR (KBr, cm⁻¹): 3058, 1716, 548, 524, 310.

Table 7
Crystal, data collection and structure refinement parameters for **3**, **6**, **7**, **9**, **11** and **13**

Complex	3	6	7	9	11	13
Empirical formula	C₂₅H₂₀O₂SSn	C₂₂H₂₈O₆S₂Sn	C₂₆H₂₀O₄S₂Sn	C₁₃H₂₂O₉SSn₂	C₄₇H₄₆O₄SSn₂	C₃₆H₅₂N₂O₄S₂Sn₄
Formula weight	503.16	539.25	579.23	479.75	944.28	1115.68
Crystal system	Triclinic	Triclinic	Triclinic	Monoclinic	Monoclinic	Triclinic
Space group	P$\bar{1}$	P$\bar{1}$	P$\bar{1}$	C2/c	P2₁/n	P$\bar{1}$
Unit cell dimension						
a (Å)	9.814(5)	8.76(3)	8.868(2)	12.820(5)	18.204(4)	7.4323(15)
b (Å)	10.125(5)	10.16(3)	12.138(3)	9.807(4)	9.991(2)	10.086(2)
c (Å)	12.241(6)	14.33(4)	12.318(3)	29.454(11)	26.030(6)	15.183(3)
α (°)	92.304(6)	100.06(5)	96.992(3)	90	90	99.911(3)
β (°)	111.864(6)	99.63(6)	100.824(2)	90.503(4)	107.148(3)	99.809(3)
γ (°)	94.615(6)	100.01(5)	102.045(4)	90	90	95.607(3)
Z	2	2	2	8	2	1
Absorption coefficient (mm⁻¹)	1.249	1.252	1.215	2.804	1.189	2.385
Crystal size (mm)	0.49 × 0.45 × 0.39	0.42 × 0.29 × 0.24	0.31 × 0.19 × 0.15	0.47 × 0.39 × 0.25	0.53 × 0.42 × 0.19	0.43 × 0.37 × 0.15
D_c (g cm⁻³)	1.489	1.480	1.537	1.721	1.3 87	1.691
θ range for data collection (°)	2.02–25.03	2.08–25.03	1.71–25.03	2.61–25.02	2.34–25.03	2.07–25.03
Reflections collected	5843	5776	6661	9228	22,686	5772
Unique reflections (R_int)	3905 (0.0116)	4053 (0.1090)	4383 (0.0214)	3275 (0.0301)	7929 (0.0361)	3808 (0.0157)
Data/restraints/parameters	3905/0/263	4053/10/262	4383/0/298	3275/0/163	7929/25/490	3808/0/217
Final R indices [I > 2σ(I)]	R₁ = 0.0278 wR₂ = 0.0724	R₁ = 0.0776, wR₂ = 0.0997	R₁ = 0.0385 wR₂ = 0.1064	R₁ = 0.0396 wR₂ = 0.1177	R₁ = 0.0411 wR₂ = 0.0955	R₁ = 0.0302 wR₂ = 0.0661
R indices (all data)	R₁ = 0.0336, wR₂ = 0.0776	R₁ = 0.2626 wR₂ = 0.1381	R₁ = 0.0609 wR₂ = 0.1223	R₁ = 0.0489 wR₂ = 0.1242	R₁ = 0.0771 wR₂ = 0.1148	R₁ = 0.0458 wR₂ = 0.0754

3.2.9. Me₃Sn(SC₆H₄CO₂)Me₃Sn (9)

The synthesis procedure was the same as **1**. 4-Sulfanylbenzoic acid (0.154 g, 1 mmol), sodium ethoxide (0.136 g, 2 mmol) and Me₃SnCl (0.398 g, 2 mmol), reaction time 12 h, temperature 40 °C. Recrystallized from ether. Colorless crystal was formed. Yield: 80%. m.p. 128–130 °C. *Anal*. Calc. for $C_{13}H_{22}O_2SSn_2$: C, 32.54; H, 4.62. Found: C, 32.55; H, 4.59%. ^{1}H NMR (CDCl₃, ppm): δ 7.15–7.40 (m, 4H), 0.70–0.75 (m, 18H). IR (KBr, cm^{-1}): 1665, 1465, 547, 522, 313.

3.2.10. (n-Bu)₃Sn(SC₆H₄CO₂)(n-Bu)₃Sn (10)

The synthesis procedure was the same as **1**. 4-Sulfanylbenzoic acid (0.154 g, 1 mmol), sodium ethoxide (0.136 g, 2 mmol) and (*n*-Bu)₃SnCl (0.652 g, 2 mmol), reaction time 12 h, temperature 40 °C. Recrystallized from ether–petroleum. Colorless crystal was formed. Yield: 76%. m.p. 83–85 °C. *Anal*. Calc. for $C_{31}H_{58}O_2SSn_2$: C, 50.85; H, 7.98. Found: C, 50.90; H, 7.99%. ^{1}H NMR (CDCl₃, ppm): δ 7.15–7.28 (m, 4H), 0.83–1.61 (m, 54H). IR (KBr, cm^{-1}): 1628, 1438, 557, 528, 316.

3.2.11. Ph₃Sn(SC₆H₄CO₂)Ph₃Sn·2EtOH (11)

The synthesis procedure was the same as **1**. 4-Sulfanylbenzoic acid (0.154 g, 1 mmol), sodium ethoxide (0.136 g, 2 mmol) and Ph₃SnCl (0.770 g, 2 mmol), reaction time 12 h, temperature 45 °C. Recrystallized from ether. Colorless crystal was formed. Yield: 85%. m.p. 186–188 °C. *Anal*. Calc. for $C_{47}H_{46}O_4SSn_2$: C, 59.78; H, 4.91. Found: C, 59.75; H, 4.99%. ^{1}H NMR (CDCl, ppm): δ 7.16–7.38 (m, 4H,C₆H₄), 7.44–7.69 (m, 30H). IR (KBr, cm^{-1}): 1634, 1477, 562, 530, 310.

3.2.12. (PhCH₂)₃Sn(SC₆H₄CO₂)(PhCH₂)₃ (12)

The synthesis procedure was the same as **1**. 4-Sulfanylbenzoic acid (0.154 g, 1 mmol), sodium ethoxide (0.136 g, 2 mmol) and (PhCH₂)₃SnCl (0.854 g, 2 mmol), reaction time 12 h, temperature 40 °C. Recrystallized from dichloromethane. Colorless crystal was formed. Yield: 70%. m.p. 180–182 °C. *Anal*. Calc. for $C_{49}H_{46}O_2SSn_2$: C, 62.85; H, 4.95. Found: C, 62.77; H, 4.98%. ^{1}H NMR (CDCl₃, ppm): δ 7.42 (m, 4H), 6.85–7.13 (m, 30H), 2.78 (s, 12H). IR (KBr, cm^{-1}): 1638, 1488, 565, 537, 309.

3.2.13. [Me₃Sn(SC₆H₄COO)Me₃Sn]₂·(4,4′-bipy) (13)

A large excess of 4,4′-bipy (0.31 g, 2.0 mmol) was added to a solution of **9** (0.478 g, 1.0 mmol) in diethyl ether (40 ml). After stirring for 5 h at room temperature, the colorless solution was concentrated and stored at low temperature to yield the product as colorless crystalline solid. Yield: 80%. m.p. 104–106 °C. *Anal*. Calc. for $C_{36}H_{52}N_2O_4S_2Sn_4$: C, 38.75; H, 4.70. Found: C, 38.77; H, 4.68%. ^{1}H NMR (CDCl₃, ppm): δ 7.55 (m, 8H) 7.28 (m, 8H), 6.85–7.13 (m, 36H), 0.68–0.77 (s, 36H). IR (KBr, cm^{-1}): 1630, 1465, 562, 530, 314.

3.3. X-ray crystallography

Crystals were mounted in Lindemann capillaries under nitrogen. All X-ray crystallographic data were collected on a Bruker SMART CCD 1000 diffractometer with graphite monochromated Mo Kα radiation ($\lambda = 0.71073$ Å) at 298(2) K. A semi-empirical absorption correction was applied to the data. The structure was solved by direct methods using SHELXL-97 and refined against F^2 by full-matrix least squares using SHELXL-97. Hydrogen atoms were placed in calculated positions. Crystal data and experimental details of the structure determinations are listed in Table 7.

4. Supplementary material

Crystallographic data (excluding structure factors) for the structure analysis of the compounds have been deposited with the Cambridge Crystallographic Data Center, CCDC No. 260185 **3**, 260183 **6**, 260182 **7**, 260181 **9**, 260184 **11** and 260186 **13**. Copies of these information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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