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Structural chemistry of organotin carboxylates

VI *. Characterization of $\{[R_2Sn(O_2CR')]_2O\}_2$ (R = Me, Et, ^nPr and ^nBu; R′ = thiophene and furan). X-ray crystal structure of $\{[^nBu_2Sn(O_2CC_4H_3S)]_2O\}_2$

C. Vatsa, V.K. Jain, T.K. Das

Chemistry Division, Bhabha Atomic Research Centre, Trombay, Bombay-400085 (India)

and E.R.T. Tiekink

Jordan Laboratories, Department of Physical and Inorganic Chemistry, University of Adelaide, Adelaide, South Australia 5001 (Australia)

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Abstract

The preparation and spectroscopic characterization of $\{[R_2Sn(O_2CR')]_2O\}_2$ (R = Me, Et, ^nPr, and ^nBu; R′ = thiophene and furan) are reported. On the basis of spectroscopic evidence (IR and NMR) the compounds were judged to adopt the usual dicarboxylato tetraorganostannoxane structural type, and there was no indication of bonding between tin and the ring S or O atoms. This was confirmed by X-ray crystallography in the case of $\{[^nBu_2Sn(O_2CC_4H_3S)]_2O\}_2$.

Introduction

Previous papers in this series have reported the synthesis and X-ray crystal structure determinations of organotin carboxylates in which the R′ group of the carboxylic acid function has an additional potential donor atom available for coordination to tin [1,2]. For the dicarboxylato tetraorganodistannoxane compound $\{[^nBu_2Sn(O_2CC_5H_4N)]_2O\}_2$, where the carboxylate ligand is derived from 2-pyridine carboxylic acid, it was demonstrated that the pyridine-N atoms were coordinated the Sn atom, leading to a new structural type for such compounds [3]. We report here the preparation, spectroscopic characterization, and, in one case, an X-ray crystal structure determination, of analogous compounds in which the

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[*] For part V see ref. 1.

carboxylate ligands are derived from thiophene-2-carboxylic acid and furan-2-carboxylic acid.

Results and discussion

Reactions of diorganotin oxide with the heterocyclic carboxylic acids, thiophene-2-carboxylic acid and furan-2-carboxylic acid in 1 : 1 stoichiometry afford complexes of the type $\{[R_2Sn(O_2CR')]_2O\}_2$. These $\{[R_2Sn(O_2CR')]_2O\}_2$ complexes can also be obtained by treatment of $[(R_2SnCl)_2O]$ with the free acid in the presence of triethylamine. The colourless, air-stable solids are obtained in good yields. Physical data are summarized in Table 1.

The assignments of infrared (IR) bands for the eight complexes were made by comparison with the IR spectra of the free acids, their sodium salts, and similar organotin compounds [4,5]. The ν_{asym} and ν_{sym} bands were assigned to the regions 1605–1645 cm^{-1} and 1300–1345 cm^{-1}, respectively. The magnitude of $\Delta\nu$ $[\nu_{asym}(COO) - \nu_{sym}(COO)]$, -300 cm^{-1}, indicates that the carboxylate ligands function as monodentate ligands under the conditions employed [6,7]. A strong band is observed in the region 635–650 cm^{-1} and is assigned to vibrations associated with the Sn–O–Sn stretch [6,7]. Stretching vibrations associated with Sn–O and Sn–C are tentatively assigned to the regions 490–650 and 570–650 cm^{-1}, respectively.

The ^1H NMR spectra of the compounds showed the expected integration and peak multiplicities; see Table 2. The Et, ^nPr and ^nBu groups attached to the Sn atom exhibited multiplets or broad resonances. For the R = Me compounds, two methyl resonances are observed which show different $^2J(^{119}Sn–H)$ values, as expected for dimeric dicarboxylato tetramethyldistannoxanes (Table 2). The high field methyl resonance with the smaller $^2J(Sn–H)$ is assigned to the exocyclic Me_2Sn

Table 1

Physical and analytical data for $\{[R_2Sn(O_2CR')]_2O\}_2$

Compound	Recrystallization solvent (% yield)	m.p. (°C)	Analysis Found (calc.) (%)		
			C	H	Sn
$\{[Me_2Sn(O_2CC_4H_3S)]_2O\}_2$	chloroform/hexane	239	29.16	3.04	43.00
	(57)		(29.61)	(3.19)	(41.81)
$\{[Et_2Sn(O_2CC_4H_3S)]_2O\}_2$	chloroform/hexane	235	35.12	4.28	38.23
	(51)		(34.65)	(4.20)	(38.05)
$\{[^nPr_2Sn(O_2CC_4H_3S)]_2O\}_2$	benzene/hexane	150	38.60	4.86	34.30
	(87)		(38.86)	(5.04)	(34.91)
$\{[^nBu_2Sn(O_2CC_4H_3S)]_2O\}_2$	benzene/hexane	156	42.46	6.11	32.00
	(75)		(42.42)	(5.75)	(32.25)
$\{[Me_2Sn(O_2CC_4H_3O)]_2O\}_2$	chloroform/hexane	245	30.87	3.04	44.34
	(70)		(31.39)	(3.39)	(44.31)
$\{[Et_2Sn(O_2CC_4H_3O)]_2O\}_2$	chloroform/hexane	208	36.36	4.31	40.60
	(59)		(36.53)	(4.43)	(40.11)
$\{[^nPr_2Sn(O_2CC_4H_3O)]_2O\}_2$	benzene/hexane	143–145	39.99	4.97	36.00
	(65)		(40.78)	(5.29)	(36.64)
$\{[^nBu_2Sn(O_2CC_4H_3O)]_2O\}_2$	benzene/hexane	123–126	44.86	6.02	33.70
	(73)		(44.36)	(6.01)	(33.72)

Table 2

^1H NMR data for $\{[R_2Sn(O_2CR')]_2O\}_2$ recorded in $CDCl_3$ solution at room temperature

Compound	δ(Sn-R) [a] (ppm)	δ (ligand protons) in ppm
$HO_2CC_4H_3S$		7.90 (d, 3.6 Hz) H-3; 7.65 (d, 4.9 Hz) H-5; 7.14 (dd, 3.9, 4.8 Hz) H-4; 9.69 (s) OH
$\{[Me_2Sn(O_2CC_4H_3S)]_2O\}_2$	1.06 (85) 0.99 (80)	7.72 (d, 3.5 Hz) H-3; 7.52 (d, 4.1 Hz) H-5; 7.10 (dd, 3.9, 4.9 Hz) H-4
$\{[Et_2Sn(O_2CC_4H_3S)]_2O\}_2$	0.41–2.26(m)	7.72 (d, 3.0 Hz) H-3; 7.49 (d, 3.0 Hz) H-5; 7.08 (dd) H-4
$\{[^nPr_2Sn(O_2CC_4H_3S)]_2O\}_2$	1.05 (t, br), 1.71 br	7.74 (d) H-3; 7.51 (d) H-5; 7.09 (dd) H-4
$\{[^nBu_2Sn(O_2CC_4H_3S)]_2O\}_2$	0.91 (t, br), 1.33 (m), 1.70 (br)	7.72 (d) H-3; 7.51 (d) H-5; 7.09 (dd) H-4
$HO_2CC_4H_3O$		7.62 (dd, 0.8, 1.7 Hz) H-3; 7.30 (dd, 0.8, 3.5 Hz) H-5; 6.52 (dd, 1.7, 3.5 Hz)
$\{[Me_2Sn(O_2CC_4H_3O)]_2O\}_2$	1.01 (90), 0.98 (80)	7.61 (s, br) H-3; 7.14 (d, 3.2 Hz) H-5; 6.53 (dd) H-4
$\{[Et_2Sn(O_2CC_4H_3O)]_2O\}_2$	0.43–2.21 (m)	7.58 (s, br) H-3; 7.11 (s, br) H-5; 6.48 (d) H-4
$\{[^nPr_2Sn(O_2CC_4H_3O)]_2O\}_2$	0.96 (t), 1.58 (m, br)	7.55 (s, br) H-3; 7.10 (s, br) H-5; 6.44 (dd) H-4
$\{[^nBu_2Sn(O_2CC_4H_3O)]_2O\}_2$	0.8 (t), 1.24 (m) 1.61 (s, br)	7.55 (s, br) H-3; 7.08 (d, 3.2 Hz) H-5; 6.47 (dd) H-4

[a] $^2J(^{119}Sn-^1H)$ in Hz.

group and the low field resonance, with the higher ^2J value, to the endocyclic Me_2Sn moiety [8–10].

The ^{13}C NMR spectra (Table 3) displayed two sets of R–Sn resonances, as expected for dimeric dicarboxylato tetraorganodistannoxanes, with high field resonances for exocyclic R_2Sn carbon atoms and downfield resonances for the endocyclic R_2Sn atoms. For the C-3 and C-4 resonances in the $^nPr_2Sn$ and $^nBu_2Sn$ complexes the separation between the two resonances were too small to be resolved. Ligand carbon atoms appeared as singlets in all cases. Owing to the low intensity of Sn–R carbon resonances in some cases, a poor signal/noise ratio prevented determination of $^1J(^{119}Sn-^{13}C)$. When they could be determined the values of $^1J(^{119}Sn-^{13}C)$ fell in the range 704–798 Hz. The C-2 carbon atom of the heterocyclic ring showed considerable (3.4–5.6 ppm) deshielding relative to those for the respective free acids. The carbon atoms C-3 and C-5 (i.e. adjacent to the hetero atom) are shielded by 1.0–3.9 and 2.2–3.5 ppm, respectively. These changes simply reflect the withdrawal of electron density from the ring to the Sn atom through the carboxylate group.

The ^{119}Sn NMR spectra displayed two well separated resonances (see Table 3) as is usually observed for dicarboxylato tetraorganodistannoxanes, and supporting the presence of a dimeric structure in solution. The low field and high field shifts observed for the distannoxanes are attributed to the exocyclic and endocyclic Sn atoms, respectively.

The spectroscopic evidence clearly favours the familiar dicarboxylato tetraorganodistannoxane structure for the compounds reported here. Importantly, in the context of this study, the spectroscopic data provide no evidence indicating coordi-

Table 3

$^{13}C\{^1H\}$ and $^{119}Sn\{^1H\}$ NMR data for $\{[R_2Sn(O_2CR')]_2O\}_2$ recorded in $CDCl_3$ solution at room temperature

Compound	δ ^{119}Sn (ppm)	δ Sn–R (ppm)	δ (ligand carbons) (ppm)				
			COO	C-2	C-3	C-4	C-5
$HO_2CC_4H_3S$			167.6	132.6	133.8	127.8	134.8
$\{[Me_2Sn(O_2CC_4H_3S)]_2O\}_2$	−171.9 −180.9	10.2 (798), 7.0 (773)	168.2	137.6	132.8	127.8	131.6
$\{[Et_2Sn(O_2CC_4H_3S)]_2O\}_2$	−208.6 −209.0	23.0, 20.8 ($Sn-CH_2-$) 10.2, 9.9 (Sn–C–Me)	168.2	137.9	132.7	127.8	131.6
$\{[^nPr_2Sn(O_2CC_4H_3S)]_2O\}_2$	−209.8 −213.8	32.7 (742), 31.1 (711) 19.1, 18.8, 18.3	168.0	138.0	132.5	127.4	131.4
$\{[^nBu_2Sn(O_2CC_4H_3S)]_2O\}_2$	−208.4 −210.4	30.6, 28.4, 27.8 27.4, 26.8, 13.6	167.9	138.2	132.4	127.7	131.3
$HO_2CC_4H_3O$			163.8	143.9	120.2	112.3	147.5
$\{[Me_2Sn(O_2CC_4H_3O)]_2O\}_2$	−162.9 −194.8	10.4, 6.1	163.3	147.3	117.0	111.9	145.3
$\{[Et_2Sn(O_2CC_4H_3O)]_2O\}_2$	−194.2 −221.3	22.8 (755), 19.5 (704) 9.9, 9.5	163.3	147.5	116.7	111.8	145.1
$\{[^nPr_2Sn(O_2CC_4H_3O)]_2O\}_2$	−196.5 −226.5	32.5, 29.7, 18.6 18.6, 17.9	163.2	147.4	116.6	111.8	145.0
$\{[^nBu_2Sn(O_2CC_4H_3O)]_2O\}_2$	−194.1 −222.4	29.7, 27.2, 26.9 26.4	162.9	147.3	116.3	111.5	144.7

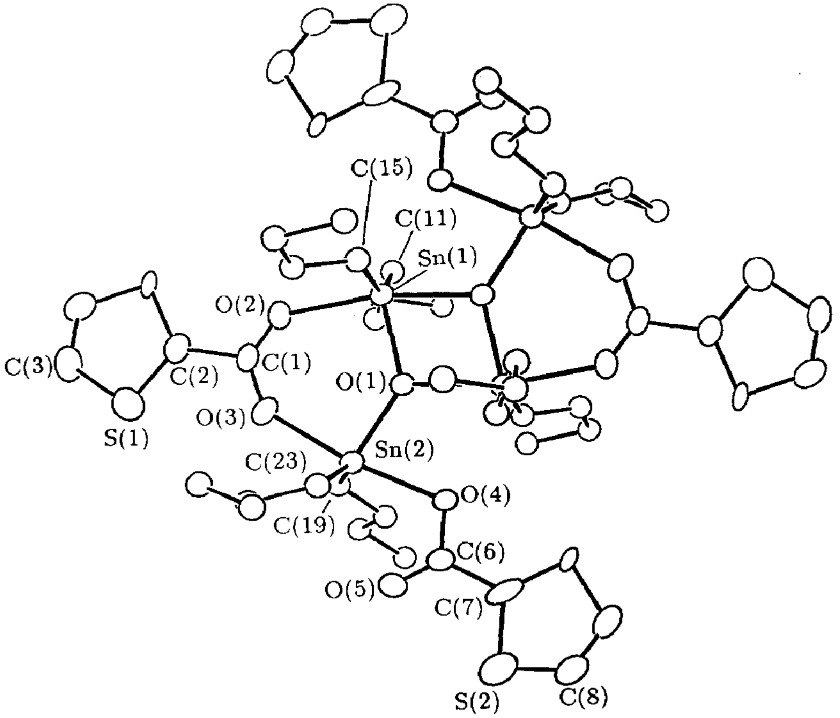

Fig. 1. Molecular structure and crystallographic numbering scheme employed for $\{[^n\text{Bu}_2\text{Sn}(\text{O}_2\text{CC}_4\text{H}_3\text{S})]_2\text{O}\}_2$.

nation of the thienyl sulphur or furanyl O atom to tin. This was confirmed by an X-ray diffraction study in the case of $\{[^n\text{Bu}_2\text{Sn}(\text{O}_2\text{CC}_4\text{H}_3\text{S})]_2\text{O}\}_2$.

The molecular structure of $\{[^n\text{Bu}_2\text{Sn}(\text{O}_2\text{CC}_4\text{H}_3\text{S})]_2\text{O}\}_2$ is shown in Fig. 1 and selected geometrical parameters for this compound are listed in Table 4. The structure determination confirms the conclusion from spectroscopic studies in it establishes the dicarboxylato tetraorganostannoxane formulation for the thiophene-2-carboxylate derivative.

The molecule is situated about a crystallographic centre of inversion and thus only half of the structure comprises the asymmetric unit. Although not of particularly high accuracy, the structure determination provides reliable evidence of the structure adopted in the solid state. The $\{[^n\text{Bu}_2\text{Sn}(\text{O}_2\text{CC}_4\text{H}_3\text{S})]_2\text{O}\}_2$ compound features a central Sn_2O_2 unit two further Sn atoms of which are coordinated via the bridging O atoms. A further link between the exocyclic and endocyclic Sn atoms is provided by two bridging carboxylate ligands (containing the O(2) and O(3) atoms). The second unique carboxylate ligand in the structure is coordinated to the Sn(2) atom through the O(4) atom only, i.e. essentially in the monodentate mode (see below). The Sn(1) and Sn(2) atoms may be regarded as five-coordinate, and to have distorted trigonal bipyramidal geometries. However, a consideration of the intramolecular Sn to O contacts shows this to be an oversimplification.

To a first approximation the coordination geometry about the Sn(1) atom would be best described as being based on a trigonal bipyramid. In this description the apical positions would be occupied by the O(1′) and O(2) atoms such that the O(1′)–Sn(1)–O(2) angle is 168.4(3)°. The trigonal plane would be defined by the

Table 4

Selected geometrical parameters (Å; deg) for $\{[^n\mathrm{Bu}_2\mathrm{Sn}(\mathrm{O}_2\mathrm{CC}_4\mathrm{H}_3\mathrm{S})]_2\mathrm{O}\}_2$

Sn(1)–O(1)	2.034(7)	Sn(1)–O(1′)	2.177(7)
Sn(1)–O(2)	2.275(9)	Sn(1)–C(11)	2.14(1)
Sn(1)–C(15)	2.08(2)	Sn(2)–O(1)	2.023(8)
Sn(2)–O(3)	2.234(9)	Sn(2)–O(4)	2.165(8)
Sn(2)–C(19)	2.12(1)	Sn(2)–C(23)	2.05(1)
Sn(1)–O(4)	2.830(8)	Sn(2)–O(5)	2.867(9)
Sn(1)–Sn(1′)	3.314(1)		
O(1)–Sn(1)–O(1′)	76.2(3)	O(1)–Sn(1)–O(2)	92.2(3)
O(1)–Sn(1)–O(4)	140.9(3)	O(1)–Sn(1)–C(11)	108.8(4)
O(1)–Sn(1)–C(15)	107.6(4)	O(1′)–Sn(1)–O(2)	168.4(3)
O(1′)–Sn(1)–O(4)	64.7(3)	O(1′)–Sn(1)–C(11)	96.0(4)
O(1′)–Sn(1)–C(15)	92.5(4)	O(2)–Sn(1)–O(4)	126.9(3)
O(2)–Sn(1)–C(11)	87.0(4)	O(2)–Sn(1)–C(15)	91.6(5)
O(4)–Sn(1)–C(11)	76.2(3)	O(4)–Sn(1)–C(15)	75.7(3)
C(11)–Sn(1)–C(15)	143.6(5)	O(1)–Sn(2)–O(3)	90.8(3)
O(1)–Sn(2)–O(4)	81.4(3)	O(1)–Sn(2)–O(5)	129.7(3)
O(1)–Sn(2)–C(19)	109.5(4)	O(1)–Sn(2)–C(23)	109.8(4)
O(3)–Sn(2)–O(4)	172.1(3)	O(3)–Sn(2)–O(5)	139.4(3)
O(3)–Sn(2)–C(19)	80.2(4)	O(3)–Sn(2)–C(23)	89.9(4)
O(4)–Sn(2)–O(5)	48.4(3)	O(4)–Sn(2)–C(19)	101.1(4)
O(4)–Sn(2)–C(23)	93.9(4)	O(5)–Sn(2)–C(19)	87.0(4)
O(5)–Sn(2)–C(23)	75.4(4)	C(19)–Sn(2)–C(23)	139.6(4)
Sn(1)–O(1)–Sn(1′)	103.8(2)		

O(1) atom and the two n-butyl groups with angles 108.9(4), 108.6(4) and 143.6(4)°. The considerable distortion in the coordination geometry about the Sn(1) atom is manifested in the large C–Sn–C angle, which arises as a result of the presence of the O(4) atom at 2.830(8) Å from the Sn(1) atom. This bond distance is approximately 0.5 Å longer than the other Sn(1)–O interactions, but if the O(4) is included in the Sn(1) coordination sphere it would occupy a position in an highly distorted octahedral geometry; see Table 4. In this description the four O atoms occupy the basal plane and the two n-butyl groups occupy the apical positions with an C–Sn–C angle of 143.6(4)°.

A similar situation exists for the Sn(2) atom. Again, to a first approximation the coordination geometry is based on a trigonal bipyramid, with an apical angle, O(3)–Sn–O(4), of 172.1(3)°; the trigonal angles range from 109.8(3) to 139.6(4)°. The larger angle can be related to the presence of the O(5) atom at 2.867(9) Å from the Sn(2) atom. Thus once again, in the light of the relatively close approach of the O(5) atom the coordination polyhedron about the Sn(2) atom can be regarded as distorted octahedral, with an O_4 basal plane.

Given that the Sn(1)–O(4) and Sn(2)–O(5) bond distances are so much longer than the other Sn–O bonds in the structure and that the sum of the covalent radii for Sn and O is 2.14 Å it is appropriate to describe the coordination polyhedra about the two Sn atoms as being trigonal bipyramidal with distortions towards the octahedral arising as the result of the close approach of non-coordinating O atoms. A similar situation was reported for the tetrahedral–trigonal bipyramidal dichotomy for compounds of the type $[\mathrm{Ph}_3\mathrm{Sn}(\mathrm{O}_2\mathrm{CR}')]$ [11,12].

The structure reported here for $\{[^nBu_2Sn(O_2CC_4H_3S)]_2O\}_2$ is similar to that reported for other dicarboxylato tetraorganostannoxane compounds that have been structurally characterized [2,13–17]. Notable in the structure, however, is the absence of a significant intra- or inter-molecular interaction between the thiophene S atom and the Sn atoms. This is in accord with data for the Me_3Sn [1] and Ph_3Sn [18] derivatives with thiophene-2-carboxylate, in which significant Sn to S interactions were absent. So far in the structural chemistry of organotin carboxylates only in compounds which have an heterocyclic N atom available for coordination to Sn has such coordination been noted.

Experimental

Preparation of compounds

The carboxylic acids, thiophene-2-carboxylic acid and furan-2-carboxylic acid and R_2SnO were obtained from Fluka. Analytical grade solvents were dried before use.

The IR spectra were recorded as Nujol mulls, in Fluorolube or as KBr pellets on a Perkin Elmer 577, 7738 and on a Matason FT-IR spectrometers. The ^1H, ^{13}C and ^{119}Sn NMR spectra were recorded on a Varian FT-80A spectrometer in $CDCl_3$ solution operating at 79.5, 20.0 and 29.6 MHz respectively. Chemical shifts are reported in ppm from an internal chloroform peak (7.26 ppm for ^1H and 77.0 for ^{13}C) and from external 33% Me_4Sn in C_6D_6 for ^{119}Sn. Tin was determined as SnO_2, and microanalyses for C and H were performed by the Analytical Chemistry Division, B.A.R.C.

Table 5

Crystal data and refinement details for $\{[^nBu_2Sn(O_2CC_4H_3S)]_2O\}_2$

Formula	$C_{52}H_{84}O_{11}S_4Sn_4$
Mol. wt.	1488.2
Crystal system	monoclinic
Space group	$P2_1/n$
a (Å)	13.756(2)
b (Å)	17.364(3)
c (Å)	13.741(2)
β (°)	93.56(2)
V (Å^3)	3275.8
Z	2
D_c (g cm^{-3})	1.509
$F(000)$	1496
μ (cm^{-1})	15.47
θ limits (°)	1.5–25.0
No. of data collected	7943
No. of unique data	6435
No. of unique reflections used with $I \geq 2.5\sigma(I)$	3393
R	0.063
k	2.65
g	0.0012
R_w	0.073

Preparation of $\{[^n\mathrm{Bu}_2\mathrm{Sn}(\mathrm{O}_2\mathrm{CC}_4\mathrm{H}_3\mathrm{S})]_2\mathrm{O}\}_2$. Method A. To a benzene solution of $\mathrm{Bu}_2\mathrm{SnO}$ (2.06 g, 8.28 mmol) was added a benzene solution of (60 cm^3) thiophene-2-carboxylic acid (1.06 g; 8.29 mmol). The mixture was refluxed for 2 h, the water formed being removed azeotropically with a Dean and Stark assembly. The clear solution obtained was evaporated under reduced pressure to leave a white solid, which was recrystallized as specified in Table 1.

Method B. To a benzene solution (20 cm^3) of $[(^n\mathrm{Bu}_2\mathrm{SnCl})_2\mathrm{O}]$ (2.00 g; 3.62 mmol) were added triethylamine (0.73 g; 7.23 mmol) and a benzene solution of thiophene-2-carboxylic acid (0.93 g; 7.24 mmol). The mixture was refluxed with stirring for 2 h. Then cooled, and triethylamine hydrochloride was filtered off and the filtrate concentrated under vacuum to yield crystalline $\{[^n\mathrm{Bu}_2\mathrm{Sn}(\mathrm{O}_2\mathrm{CC}_4\mathrm{H}_3\mathrm{S})]_2\mathrm{O}\}_2$. The other compounds were prepared by these methods.

Table 6

Fractional atomic coordinates ($\times 10^4$) for $\{[^n\mathrm{Bu}_2\mathrm{Sn}(\mathrm{O}_2\mathrm{CC}_4\mathrm{H}_3\mathrm{S})]_2\mathrm{O}\}_2$

Atom	*x*	*y*	*z*
Sn(1)	5328(1)	148(1)	1166(1)
Sn(2)	2739(1)	422(1)	505(1)
S(1)	9547(5)	112(5)	2362(5)
S(2)	2631(6)	940(5)	4466(4)
O(1)	5907(5)	−179(4)	−96(5)
O(2)	6787(7)	25(6)	2027(7)
O(3)	7879(9)	−241(11)	1020(9)
O(4)	3533(6)	588(5)	1900(5)
O(5)	2091(7)	767(8)	2400(7)
C(1)	7600(11)	−50(10)	1824(10)
C(2)	8404(11)	153(8)	2598(9)
C(3)	9925(14)	345(13)	3413(13)
C(4)	9244(17)	490(12)	4012(12)
C(5)	8168(8)	434(7)	3629(7)
C(6)	2918(10)	698(7)	2582(9)
C(7)	3420(14)	784(7)	3609(9)
C(8)	6469(15)	−949(12)	−5312(11)
C(9)	5575(17)	−919(11)	−4987(11)
C(10)	4519(7)	788(6)	3846(6)
C(11)	4873(9)	−858(7)	1914(9)
C(12)	5503(12)	−1572(9)	1806(13)
C(13)	5163(16)	−2243(13)	2422(17)
C(14)	4382(28)	−2707(24)	1835(31)
C(15)	5401(11)	1342(10)	1243(11)
C(16)	6279(19)	1789(15)	1550(24)
C(17)	6284(21)	2670(15)	1600(22)
C(18)	5326(33)	2816(39)	1051(40)
C(19)	7623(15)	−1573(12)	−122(16)
C(20)	7322(16)	−2228(13)	−774(17)
C(21)	7772(19)	−2972(14)	−349(18)
C(22)	7441(23)	−3643(17)	−952(23)
C(23)	7923(18)	567(13)	−929(18)
C(24)	8921(27)	842(35)	−587(41)
C(25)	9325(59)	1635(31)	−261(35)
C(26)	8890(23)	1932(20)	−1217(24)

Crystallography

Intensity data were measured at room temperature on an Enraf-Nonius CAD4F diffractometer fitted with graphite monochromatized Mo-K_α radiation, $\lambda = 0.7107$Å. The ω–2θ scan technique was employed to measure 7943 reflections up to a maximum Bragg angle of 25.0°. The data were corrected for Lorentz and polarization effects but not for absorption. Relevant crystal data are collected in Table 5.

The structure was solved by direct methods [19] and refined by a full-matrix least-squares procedure based on F[19]. Owing to significant disorder and high thermal motion the butyl groups were refined with constrained C–C bond distances (1.52(2) Å) and isotropic thermal parameters; the remaining atoms were refined anisotropically. Hydrogen atoms were not included in the model. After the inclusion of a weighting scheme of the form, $w = k/[\sigma^2(F) + g\,|F|^2]$, the refinement was continued until convergence. Final refinement details are listed in Table 5. The analysis of variance showed no special features. Fractional atomic coordinates are listed in Table 6 and the numbering scheme employed is shown in Fig. 1 drawn with ORTEP [20] at 15% probability ellipsoids. Scattering factors were as incorporated in the SHELX76 program [19] and refinement was performed on a SUN4/280 computer. Other crystallographic details including thermal parameters, a complete list of bond distances and angles, and lists of observed and calculated structure factors, are available from E.R.T.T.

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