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## Diaryloxytetraorganodistannoxanes: synthesis and characterization by IR and NMR ( $^1\text{H}$ , $^{13}\text{C}$ and $^{119}\text{Sn}$ ) spectroscopy. Crystal structure of $\{[{}^n\text{Bu}_2\text{Sn}(\text{OC}_6\text{H}_4\text{OMe-2})]_2\text{O}\}_2$

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### Abstract

Diaryloxytetraorganodistannoxanes,  $\{[\text{R}_2\text{Sn}(\text{OAr})]_2\text{O}\}_2$  ( $\text{R} = \text{Me, Et, Pr, Bu}$ ;  $\text{OAr} = \text{OPh, OC}_6\text{H}_4\text{Cl-2, OC}_6\text{H}_4\text{Me-2, OC}_6\text{H}_4\text{OMe-2, OC}_6\text{H}_4\text{OEt-2}$ ) have been prepared and characterized by IR and NMR ( $^1\text{H}$ ,  $^{13}\text{C}$ ,  $^{119}\text{Sn}$ ) spectroscopy. The compounds are assigned tetranuclear distannoxane structures in solution. The crystal structure of a representative compound,  $\{[{}^n\text{Bu}_2\text{Sn}(\text{OC}_6\text{H}_4\text{OMe-2})]_2\text{O}\}_2$ , has confirmed the spectroscopic assignments. The centrosymmetric molecule features a planar  $\text{Sn}_2\text{O}_2$  unit with a  ${}^n\text{Bu}_2\text{Sn}$  group connected to each bridging O atom. There are two independent  $\text{OC}_6\text{H}_4\text{OMe-2}$  ligands in the structure, one bridges both the *endo*- and *exo*-cyclic Sn atoms and the other is coordinated only to the *exo*-cyclic Sn atom; the result is that both Sn atoms are five-coordinate, and have distorted trigonal bipyramidal geometries.

### Introduction

Diorganotin compounds, particularly tetraorganodistannoxanes, are of current interest in view of their applications as catalysts in industry and as biocides [1,2]. The bonding of X ligands in tetraorganodistannoxanes,  $[\text{R}_2\text{SnX}]_2\text{O}$ , shows a range of structural variations, as revealed by X-ray structure determinations for compounds with  $\text{X} = \text{RCOO}$  [3],  $\text{Cl}$  [4] or  $\text{NCS}$  [5]. Reactions of diorganotin oxides with phenols have been described in the literature [6]; such reactions yield a variety of products, viz. hydroxo compounds  $[\text{R}_2\text{Sn}(\text{OH})(\text{OAr})]$ , tetraorganodistannoxanes  $\{[\text{R}_2\text{Sn}(\text{OAr})]_2\text{O}\}_2$ , and bis complexes,  $[\text{R}_2\text{Sn}(\text{OAr})_2]$ , depending on the substituents on the aryl group and the reaction conditions. The reactions of some substituted phenols with diorganotin oxides and the characterization of the products by IR and multinuclear magnetic resonance spectroscopy form the subject of this

report. One of the products, namely  $\{[{}^n\text{Bu}_2\text{Sn}(\text{OC}_6\text{H}_4\text{OMe-2})]_2\text{O}\}_2$ , has been fully characterized by X-ray crystallography.

## Results and discussion

Reactions of diorganotin(IV) oxides with phenols in 1:1 or 1:2 stoichiometry in refluxing benzene or toluene afforded white crystalline, air-stable complexes. These were purified by recrystallization from benzene, benzene-ether, diethyl ether, or hexane in 40–90% yields. Physical and analytical data for these complexes are listed in Table 1.

The IR absorption bands in the region  $3125\text{--}2700\text{ cm}^{-1}$ , assigned to the  $\nu(\text{OH})$  mode in the free ligands, are absent in the complexes, indicating deprotonation of the phenolic H atom. This, together with elemental analyses, established the tetraorganodistannoxane formulation for the complexes. Assignments of various vibrational modes for the complexes were based on comparison with the IR data for related compounds  $(\text{R}_2\text{SnO})_n$  and free phenols [7–12]. A band in the region  $495\text{--}530\text{ cm}^{-1}$  was assigned to  $\nu(\text{Sn}\text{--}\text{O})$  and a strong band in the region  $610\text{--}680\text{ cm}^{-1}$  to the  $\nu(\text{Sn}\text{--}\text{O}\text{--}\text{Sn})$  mode. The symmetric and asymmetric  $\nu(\text{Sn}\text{--}\text{C})$  stretchings were assigned to the region  $525\text{--}615\text{ cm}^{-1}$  and the C–O absorptions to the region  $1200\text{--}1295\text{ cm}^{-1}$ .

Table 1

Physical and analytical data for the  $\{[\text{R}_2\text{Sn}(\text{OAr})]_2\text{O}\}_2$  complexes

Complex	M.p. (°C)	Yield (%)	Analysis (Found (calc.) (%))		
			C	H	Sn
$\{[\text{Me}_2\text{Sn}(\text{OC}_6\text{H}_5)]_2\text{O}\}_2$	200	60	38.10 (38.45)	4.01 (4.43)	46.97 (47.50)
$\{[\text{Bu}_2\text{Sn}(\text{OC}_6\text{H}_5)]_2\text{O}\}_2$	134	87.5	–	–	35.34 (35.53)
$\{[\text{Pr}_2\text{Sn}(\text{OC}_6\text{H}_4\text{Cl-2})]_2\text{O}\}_2$	116	85	42.10 (42.33)	5.54 (5.32)	34.26 (34.86)
$\{[\text{Bu}_2\text{Sn}(\text{OC}_6\text{H}_4\text{Cl-2})]_2\text{O}\}_2$	121	73	–	–	31.87 (32.20)
$\{[\text{Bu}_2\text{Sn}(\text{OC}_6\text{H}_4\text{Me-2})]_2\text{O}\}_2$	liq.	98	–	–	33.82 (34.10)
$\{[\text{Me}_2\text{Sn}(\text{OC}_6\text{H}_4\text{OMe-2})]_2\text{O}\}_2$	241	–	38.45 (38.58)	4.87 (4.68)	41.87 (42.40)
$\{[\text{Et}_2\text{Sn}(\text{OC}_6\text{H}_4\text{OMe-2})]_2\text{O}\}_2$	205	40	42.90 (42.90)	5.62 (5.56)	38.01 (38.54)
$\{[\text{Pr}_2\text{Sn}(\text{OC}_6\text{H}_4\text{OMe-2})]_2\text{O}\}_2$	180	–	45.95 (46.46)	6.49 (6.30)	34.98 (35.32)
$\{[\text{Bu}_2\text{Sn}(\text{OC}_6\text{H}_4\text{OMe-2})]_2\text{O}\}_2$	170	87	49.81 (49.48)	7.22 (6.92)	32.01 (32.60)
$\{[\text{Me}_2\text{Sn}(\text{OC}_6\text{H}_4\text{OEt-2})]_2\text{O}\}_2$	208	30	39.92 (40.86)	5.70 (5.14)	39.69 (40.38)
$\{[\text{Pr}_2\text{Sn}(\text{OC}_6\text{H}_4\text{OEt-2})]_2\text{O}\}_2$	140	55	47.65 (48.03)	6.43 (6.62)	33.07 (33.91)
$\{[\text{Bu}_2\text{Sn}(\text{OC}_6\text{H}_4\text{OEt-2})]_2\text{O}\}_2$	145	90	50.02 (50.82)	7.05 (7.19)	31.28 (31.39)

Table 2

<sup>1</sup>H NMR spectral data for {[Me<sub>2</sub>Sn(OAr)]<sub>2</sub>O}<sub>2</sub>

Compound	Me <sub>2</sub> Sn resonance	Phenolic protons
{[Me <sub>2</sub> Sn(OC <sub>6</sub> H <sub>5</sub> )] <sub>2</sub> O} <sub>2</sub>	1.02 (79 Hz)	7.22 (t, 7.5 Hz)
	0.84 (75 Hz)	6.83 (t, 7.3 Hz)
		6.62 (d, 8.2 Hz)
{[Me <sub>2</sub> Sn(OC <sub>6</sub> H <sub>4</sub> OMe-2)] <sub>2</sub> O} <sub>2</sub>	0.89 (87 Hz)	3.88 (OMe)
	0.77 (83 Hz)	6.72 (d)
		6.83 (d, d)
{[Me <sub>2</sub> Sn(OC <sub>6</sub> H <sub>4</sub> OEt-2)] <sub>2</sub> O} <sub>2</sub>	0.92 (84 Hz)	1.50 (t)
	0.80 (80 Hz)	4.15 (q)
		6.69–7.24 (m)

The <sup>1</sup>H NMR spectra showed complex multiplets for aromatic protons and R<sub>2</sub>Sn groups. For the dimethyltin(IV) complexes two singlets (Table 2) were observed, indicating the presence of two types of methyl groups. A singlet was observed for the OMe group of the ligand in the spectra of both the free ligand and in the complexes.

The <sup>13</sup>C{<sup>1</sup>H} and <sup>119</sup>Sn{<sup>1</sup>H} NMR spectra were recorded in CDCl<sub>3</sub> solution at room temperature and the resulting data are summarized in Table 3. The <sup>13</sup>C NMR data for the complexes displayed two sets of resonances for Sn–R C atoms. In some cases C-3 and C-4 carbons of the Pr<sub>2</sub>Sn and Bu<sub>2</sub>Sn complexes appeared as singlets. The magnitude of <sup>1</sup>J(Sn–C) clearly indicates that the tin atoms have coordination numbers of five or six.

Assignments of ligand carbons are based on the reported data for phenols and related molecules [13]. The C-1 signals in all the complexes is deshielded with respect to the corresponding resonance of the free ligand. The carbon resonances of the *o*-methoxy and *o*-ethoxy groups showed little change in the complexes from their positions in the free ligands, which suggests that the ether O atoms do not participate in coordination to the Sn atoms.

The <sup>119</sup>Sn NMR spectra for these complexes exhibited either a broad resonance or two well separated signals. The <sup>119</sup>Sn NMR data for {[Bu<sub>2</sub>Sn(OPh)]<sub>2</sub>O}<sub>2</sub> are consistent with the data reported recently [14], although others [15] have reported a broad resonance at δ 181 ± 5 ppm for this compound.

It is noteworthy that the <sup>119</sup>Sn NMR shifts for the phenoxy complexes are considerably shielded compared to those of the corresponding chlorides, {[R<sub>2</sub>SnCl]<sub>2</sub>O}<sub>2</sub> (e.g. R = Bu: δ -90.2, -143.1 [14]), but are comparable to those of the corresponding fluorides (e.g. {[Bu<sub>2</sub>SnF]<sub>2</sub>O}<sub>2</sub>: δ -168 ± 14 ppm [15]). Clearly the electronegativities of the substituents contribute to the <sup>119</sup>Sn NMR chemical shifts of these complexes.

Tetraorganodistannoxanes have been found to form a dimeric ladder structure in all cases for which X-ray structure determinations have been reported. The IR and NMR spectroscopic data obtained for the compounds reported herein are consistent with such a formulation. The single crystal structure determination of one derivative, {[<sup>n</sup>Bu<sub>2</sub>Sn(OC<sub>6</sub>H<sub>4</sub>OMe-2)]<sub>2</sub>O}<sub>2</sub>, corroborates the spectroscopic evidence.

The molecular structure of {[<sup>n</sup>Bu<sub>2</sub>Sn(OC<sub>6</sub>H<sub>4</sub>OMe-2)]<sub>2</sub>O}<sub>2</sub> is shown in Fig. 1 and selected interatomic parameters are listed in Table 4. The structure adopted by this

Table 3  
Multinuclear magnetic resonance data (for the  $\{[R_2Sn(OAr)]_2O\}_2$  complexes

Complex	$\delta(^{119}Sn(^1H))$ (ppm)	$\delta(Sn-R)$ carbons <sup>a</sup> (ppm)	$^{13}C(^1H)$ NMR data—ligand carbons							
			C-1	C-2	C-3	C-4	C-5	C-6	O-substituents	
$C_6H_5OH$ <sup>b</sup>	—	—	C-1	C-2	C-3	C-4	C-5	C-6	O-substituents	
$\{[Me_2Sn(OC_6H_5)]_2O\}_2$	-138	2.4(659, 628)	155.1	115.7	130.1	121.4	130.1	115.7	—	
	-140	4.5(671, 643)	159.6	118.5	129.6	119.1	129.6	118.5	—	
$\{[Bu_2Sn(OC_6H_5)]_2O\}_2$	-176.8	C-1 24.9, 23.2	160.9	117.9	129.4	118.9	129.4	117.9	—	
	-177.4	C-2 27.3, 26.9								
		C-3 27.4, 27.1								
		C-4 13.5, 13.4								
2-ClC <sub>6</sub> H <sub>4</sub> OH	—	—	151.5	120.1	121.5	128.5	129.2	116.5	—	
$\{[Pr_2Sn(OC_6H_4Cl-2)]_2O\}_2$	-169 (broad)	C-1 29.0, 28.5	156.0	120.1	123.2	127.6	130.1	118.7	—	
		C-2 18.9								
		C-3 18.4								
$\{[Bu_2Sn(OC_6H_4Cl-2)]_2O\}_2$	-165 (broad)	C-1 26.6(599)	156.6	120.3	123.3	127.6	130.1	118.7	—	
		25.9(612)								
		C-2 27.5(120)								
		27.0(121)								
		C-3 27.3, 26.9(55)								
		C-4 13.5, 13.3								
2-MeC <sub>6</sub> H <sub>4</sub> OH <sup>b</sup>	—	—	153.1	124.0	131.0	121.4	127.7	115.9	16.7	
$\{[Bu_2Sn(OC_6H_4Me-2)]_2O\}_2$ <sup>c</sup>	-165	C-1 24.1, 23.0	156.9	126.2	130.9	119.2	128.2	117.4	16.7	
		C-2,3 27.7, 27.2								
		26.9								
		C-4 13.3								



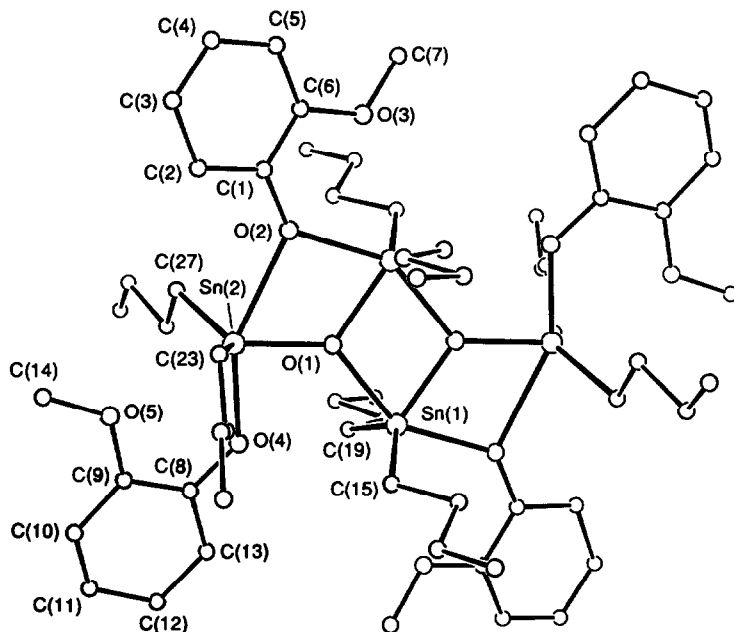


Fig. 1. Molecular structure of  $\{[{}^n\text{Bu}_2\text{Sn}(\text{OC}_6\text{H}_4\text{OMe-2})]_2\text{O}\}_2$  and crystallographic numbering scheme

Table 4

Selected interatomic parameters (Å, deg) for  $\{[{}^n\text{Bu}_2\text{Sn}(\text{OC}_6\text{H}_4\text{OMe-2})]_2\text{O}\}_2$

Sn(1)–O(1)	2.150(5)	Sn(1)–O(2)	2.198(5)
Sn(1)–O(1) <sup>a</sup>	2.038(5)	Sn(1)–C(15)	2.12(1)
Sn(1)–C(19)	2.14(1)	Sn(2)–O(1)	2.011(5)
Sn(2)–O(2)	2.470(5)	Sn(2)–O(4)	2.061(5)
Sn(2)–C(23)	2.134(8)	Sn(2)–C(27)	2.123(9)
O(2)–C(1)	1.342(8)	C(1)–C(2)	1.39(1)
C(1)–C(6)	1.37(1)	C(6)–O(3)	1.39(1)
O(3)–C(7)	1.42(2)	O(4)–C(8)	1.35(1)
C(8)–C(13)	1.36(1)	C(8)–C(9)	1.36(1)
C(9)–O(5)	1.39(1)	O(5)–C(14)	1.38(1)
O(1)–Sn(1)–O(2)	147.2(2)	O(1)–Sn(1)–O(1)′	73.3(2)
O(1)–Sn(1)–C(15)	92.8(3)	O(1)–Sn(1)–C(19)	93.9(3)
O(2)–Sn(1)–O(1)′	74.1(2)	O(2)–Sn(1)–C(15)	101.4(3)
O(2)–Sn(1)–C(19)	96.9(3)	O(1)′–Sn(1)–C(15)	110.1(3)
O(1)′–Sn(1)–C(19)	115.3(3)	C(15)–Sn(1)–C(19)	134.1(4)
O(1)–Sn(2)–O(2)	68.6(2)	O(1)–Sn(2)–O(4)	82.1(2)
O(1)–Sn(2)–C(23)	111.1(3)	O(1)–Sn(2)–C(27)	112.0(3)
O(2)–Sn(2)–O(4)	150.7(2)	O(2)–Sn(2)–C(23)	88.3(3)
O(2)–Sn(2)–C(27)	84.3(3)	O(4)–Sn(2)–C(23)	104.3(3)
O(4)–Sn(2)–C(27)	106.1(3)	C(23)–Sn(2)–C(27)	129.7(4)
Sn(1)–O(1)–Sn(2)	133.0(2)	Sn(1)–O(1)–Sn(1)′	106.7(2)
Sn(2)–O(1)–Sn(1)′	120.0(2)	Sn(1)–O(2)–Sn(2)	97.2(2)
Sn(1)–O(2)–C(1)	132.2(5)	Sn(2)–O(2)–C(1)	130.2(5)
Sn(2)–O(4)–C(8)	130.5(5)		

<sup>a</sup> Primed atoms are related by centre of inversion.

compound conforms to the familiar tetraorganodistannoxane pattern, the so-called ladder structure. The centrosymmetric molecule is constructed about the central  $\text{Sn}_2\text{O}_2$  moiety with two *exo*-cyclic Sn atoms connected at the bridging O atoms, with the result that these O atoms are tridentate. Each pair of *exo*- and *endo*-cyclic Sn atoms is bridged by the oxo function of the 2-methoxyphenolate anions. The *exo*-cyclic Sn atoms are also each coordinated by a terminal 2-methoxyphenolate anion. The bridging 2-methoxyphenolate anion forms Sn–O bonds of different lengths, viz. Sn(1)–O(2) 2.198(5) and Sn(2)–O(2) 2.470(5) Å.

Both Sn atoms are five-coordinate and exists in distorted trigonal bipyramidal geometries. The Sn(1) atom lies 0.084(1) Å out of the basal plane, defined by the C(15), C(19) and O(1)' atoms, in the direction of the O(2) atom. The distortion from the ideal geometry is greater about the Sn(2) atom owing to the longer Sn(2)–O(2) bond distance. The Sn(2) atom lies 0.324(1) Å out of the plane defined by the C(23), C(27) and O(1) atoms in the direction of the O(4) atom. The O(4) atom is 3.236(6) Å from the Sn(1) atom, a distance that does not indicate a significant bonding interaction between these atoms. It is noteworthy that the methoxy-O atoms are significantly closer to the Sn atoms. The Sn(1)···O(3) separation is 2.893(6) Å and the Sn(2)···O(5) separation is slightly shorter at 2.792(6) Å. If these interactions were considered significant, the Sn(1) and Sn(2) atoms could be thought to exist in distorted octahedral geometries with the basal planes being defined by four O atoms in each case. That the C–Sn–C angles differ greatly from the ideal octahedral angle of 180° (C(15)–Sn–C angles differ greatly from the ideal octahedral angle of 180° (C(15)–Sn(1)–C(19) is 134.1(4) and C(23)–Sn(2)–C(27) is 129.7(4)°) suggests that these Sn···O(ether) interactions should not be regarded as representing bonding interactions. The Sn atoms are therefore best described as having trigonal bipyramidal geometries. Thus the close approach of the ether O atoms to the Sn atoms does not result in a formal increase in coordination number for each Sn atom, but does influence the degree of distortion of the coordination polyhedron about each Sn atom.

## Experimental

### Synthesis

The reagents 2-methoxy- and 2-ethoxy-phenol and  $\text{Bu}_2\text{SnO}$  were obtained from Fluka. Phenol, 2-chlorophenol and 2-cresol were purified by distillation before use. Microanalyses were carried out by the Bio-organic Division, BARC and tin was estimated as tin oxide. IR spectra were recorded on a Perkin–Elmer 577 spectrometer.  $^1\text{H}$  NMR spectra were recorded on a Bruker 200 MHz NMR spectrometer.  $^{13}\text{C}$  and  $^{119}\text{Sn}$  NMR spectra were recorded on a Varian FT-80A NMR spectrometer at 20.0 and 29.6 MHz, respectively. Some of the  $^{119}\text{Sn}$  NMR spectra were recorded on a Bruker AM-500 spectrometer at 185 MHz.

### Preparation of $\{[{}^n\text{Bu}_2\text{Sn}(\text{OC}_6\text{H}_4\text{OMe-2})]_2\text{O}\}_2$

To a benzene suspension of dibutyltin oxide (3.77 g, 15.0 mmol) was added a benzene solution of 2-methoxyphenol (1.88 g, 15.1 mmol). The mixture was heated under reflux for 2 h and the water formed continuously removed azeotropically with a Dean–Stark trap. The solvent was evaporated off under vacuum to leave a white crystalline product, which was recrystallized from anhydrous diethyl ether (yield

Table 5

Crystal data and refinement details for  $\{[{}^n\text{Bu}_2\text{Sn}(\text{OC}_6\text{H}_4\text{OMe-2})]_2\text{O}\}_2$ 

Formula	$\text{C}_{60}\text{H}_{100}\text{O}_{10}\text{Sn}_4$
Mol. wt.	1456.2
Crystal system	monoclinic
Space group	$C2/c$
$a$ , Å	26.503(6)
$b$ , Å	12.522(2)
$c$ , Å	25.536(3)
$\beta$ , deg	126.74(2)
$V$ , Å <sup>3</sup>	6791.2
$Z$	4 (tetramers)
$D_c$ , g cm <sup>-3</sup>	1.424
$F(000)$	732
$\mu$ , cm <sup>-1</sup>	13.77
$T$ , K	293
No. of data collected	5629
No. of unique data	4454
No. of unique reflections used with $I \geq 2.5\sigma(I)$	2782
$R$	0.042
$k$	2.83
$g$	0.0005
$R_w$	0.047
Residual $\rho_{\text{max}}$ , e Å <sup>-3</sup>	0.48

87%, 4.79 g). Other diaryloxytetraorganodistannoxanes were prepared by the same route. However, dimethyl- or diethyl-tin oxides did not dissolve to a significant extent when treated with *o*-substituted phenols in equimolar quantities, even in high boiling point solvents like toluene. They react partially, to give the desired products, when a 1 : 2 stoichiometry was used in refluxing toluene. Pertinent data for these complexes are given in Table 1.

### Crystallography

Intensity data for  $\{[{}^n\text{Bu}_2\text{Sn}(\text{OC}_6\text{H}_4\text{OMe-2})]_2\text{O}\}_2$  were measured at 293 K on an Enraf–Nonius CAD4F diffractometer fitted with graphite monochromatized Mo- $K_\alpha$  radiation,  $\lambda = 0.7107$  Å. The  $\omega : 2\theta$  scan technique was employed to measure data up to a maximum Bragg angle of 22.5°. The data set was corrected for Lorentz and polarization effects but not for absorption owing to the spherical shape of the crystal. Relevant crystal data are given in Table 5.

The structure was solved by direct-methods [16] and refined by a full-matrix least-squares procedure based on  $F$  [17] with anisotropic thermal parameters for non-butyl and non-H atoms. Butyl atoms were refined with isotropic thermal parameters owing to high thermal motion associated with these atoms. The butyl group, labelled C(19)–C(22), was disordered over two sites; each was refined at 50% site occupancy. Hydrogen atoms were included in the model at their calculated positions except for the C(19)–C(22) atoms. After the inclusion of a weighting scheme of the form,  $w = [\sigma^2(F) + g|F|^2]^{-1}$ , the refinement was continued until convergence; final refinement details are listed in Table 5. The analysis of variance



Table 6

Fractional atomic coordinates ( $\times 10^5$  for Sn,  $\times 10^4$  for other atoms) for  $\{[{}^{119}\text{Bu}_2\text{Sn}(\text{OC}_6\text{H}_4\text{OMe-2})]_2\text{O}\}_2$ 

Atom	x	y	z
Sn(1)	30491(3)	33705(4)	5472(3)
Sn(2)	21872(3)	15165(5)	9572(3)
O(1)	2416(2)	2124(4)	396(2)
O(2)	3456(2)	4281(5)	147(3)
O(3)	3911(5)	5100(7)	1307(4)
O(4)	2900(3)	2515(5)	1644(3)
O(5)	2421(4)	1475(5)	2178(3)
C(1)	3940(4)	4967(7)	421(4)
C(2)	4209(4)	5246(7)	110(5)
C(3)	4744(4)	5944(8)	430(6)
C(4)	4976(5)	6353(8)	1004(6)
C(5)	4736(5)	6053(10)	1324(5)
C(6)	4200(5)	5400(8)	1023(5)
C(7)	4001(10)	5700(15)	1827(9)
C(8)	3145(4)	2674(7)	2276(4)
C(9)	2930(5)	2158(8)	2575(5)
C(10)	3202(6)	2349(9)	3241(5)
C(11)	3664(7)	2974(11)	3587(6)
C(12)	3927(6)	3522(10)	3316(6)
C(13)	3638(6)	3363(7)	2637(6)
C(14)	2190(7)	848(10)	2437(7)
C(15)	2521(5)	4568(8)	610(5)
C(16)	2266(7)	5493(10)	136(7)
C(17)	1932(9)	6210(13)	340(9)
C(18)	1649(11)	7055(17)	-88(11)
C(19)	3876(5)	2478(8)	1252(5)
C(20)	4356(8)	2246(14)	1123(9)
C(21)	4706(13)	1264(19)	1554(15)
C(22)	5146(16)	1145(25)	1373(18)
C(20')	3998(10)	1646(15)	942(11)
C(21')	4529(14)	826(21)	1381(23)
C(22')	5036(13)	1666(19)	1734(13)
C(23)	1350(4)	2238(7)	735(4)
C(24)	1447(5)	3350(7)	1033(6)
C(25)	778(6)	3817(11)	748(8)
C(26)	798(11)	4786(15)	1015(12)
C(27)	2531(4)	-63(7)	1269(5)
C(28)	3231(5)	-158(8)	1786(6)
C(29)	3400(7)	-1339(10)	1962(8)
C(30)	4057(8)	-1498(13)	2433(10)

showed no special features indicating that an appropriate weighting scheme had been applied. Fractional atomic coordinates are listed in Table 6 and the numbering scheme employed is shown in Fig. 1 which was drawn with ORTEP [18] using arbitrary thermal ellipsoids. Scattering factors were as incorporated in the SHELX76 program [17] and the refinement was performed on a SUN4/280 computer. Other crystallographic details (available from ERTT) comprise thermal parameters, H-atom parameters, all bond distances and angles, and tables of observed and calculated structure factors.

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