THE STRUCTURE OF 2,2-DIALKYL-1,3,2-OXATHIASTANNOLANES *

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Summary

A single crystal X-ray diffraction study of 2,2-dibutyl-1,3,2-oxathiastannolane shows that individual molecules are coordinated through oxygen to give a linear polymer containing 5-coordinate tin in a distorted trigonal bipyramidal configuration. The physical properties of the diethyl compound are similar, and it probably has a similar structure, but 2,2-dimethyl-1,3,2-oxathiastannolane is a relatively high melting, insoluble, amorphous solid, suggesting that it is more firmly associated into a polymer. These conclusions are supported by a comparison of the Mössbauer and solution and solid state NMR spectra of the three compounds.

Introduction

The dioxastannolanes (I) have attracted a lot of attention recently because of their importance in organic synthesis, and the related oxathia- (II) and dithia-stannolanes (III) provide an interesting comparison. All three classes of compound are more prone than their acyclic analogues to increase their coordination numbers from four to five or six by ligation or, particularly, by self-association, perhaps because this relieves strain in the five-membered ring. In ring-substituted dioxastannolanes, this self-association in solution can lead to regioselective reaction of the diol moiety, and this has been exploited in a number of preparative procedures [1].

The particular techniques which have been used to study the structures of these compounds in solution include ^1H, ^{13}C, and ^{119}Sn NMR spectroscopy. The nature and degree of self association may be enhanced in the solid state. Here ^{119m}Sn Mössbauer spectroscopy has been used extensively to study structure; the first

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* Dedicated to Professor G.E. Coates on the occasion of his seventieth birthday.

measurement of a dioxastannolane by solid state high resolution ^{119}Sn NMR spectroscopy has recently been reported [2], and three dioxastannolanes and one dithiastannolane have been subjected to single crystal X-ray diffraction.



(I)   (II)   (III)
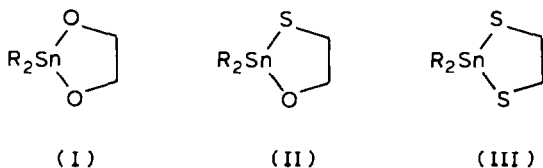
The main conclusions which have been reached are as follows.

The dioxastannolanes (I) exist in solution principally as the dimers (IV) containing 5-coordinate tin [3]. In the solid state, the degree of association depends on the structure of the groups R and of the diol moiety. Single crystal X-ray diffraction has shown that 2,2-dibutyl-1,3,2-dioxastannolane itself is an infinite ribbon polymer (V) containing 6-coordinate tin [2], but the dibutylstannyl derivative of methyl 4,6-di-O-benzylidene-α-D-mannopyranoside is a pentamer [4], and the corresponding glucopyranoside is a simple dimer [5]. 2,2-Di-t-butyl-1,3,2-dioxastannolane has also recently been shown to have a dimeric structure (IV; R = Bu[t]) [6].


(IV)   (V)
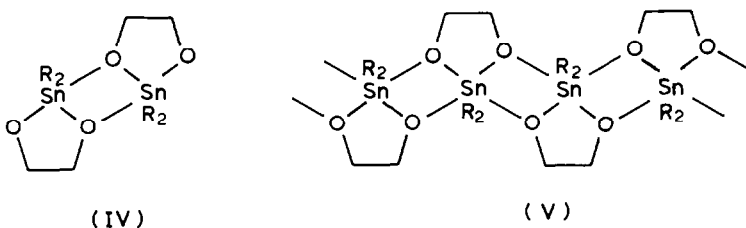
The dithiastannolanes (III) which have been studied are monomeric in solution [7–9] but in the crystal, 2,2-dimethyl-1,3,2-dithiastannolane (III, R = Me) exists as a linear polymer (VI) containing 5-coordinate tin in a distorted trigonal bipyramidal environment [10,11].


(VI)
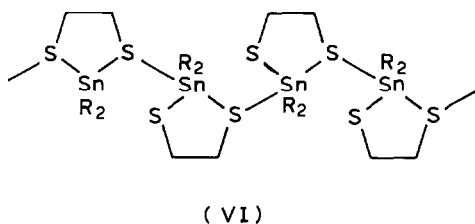
Relatively little work has been carried out on the oxathiastannolanes (II) which are our concern in this paper. The derivatives (II), R = Me [8,12,13], Et [8,14], Pr [15], Bu [8,14,16–18], Oct [17], Ph [13,18], and PhCH$_2$ [15,18] have been prepared. When R = Me, Pr, Bu, and PhCH$_2$ [15,18] colligative measurements show the compounds (II) to be dimeric in solution, and the [119]Sn chemical shift values of ca. − 30 ppm indicate that the compounds contain 5-coordinate tin in the structure (VII). From a detailed study of the temperature-dependent Mössbauer spectrum, Hager et al. [13] concluded that II, (R = Me) in the solid state had the structure of a

linear polymer (VIII) in which trigonal bipyramidal tin atoms are apically bridged through oxygen.
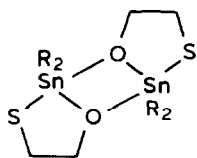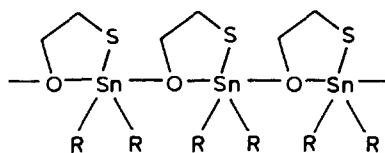
(VII)   (VIII)

We report here a study of the structures of 2,2-dimethyl-, 2,2-diethyl-, and 2,2-dibutyl-1,3,2-oxathiastannolane by a variety of techniques which include solution and solid state ^{119}Sn NMR spectroscopy, Mössbauer spectroscopy, and single crystal X-ray diffraction.

Discussion

2,2-Dimethyl-, 2,2-diethyl-, and 2,2-dibutyl-1,3,2-oxathiastannolane (II) (R = Me, Et, or Bu) were prepared by azeotropic dehydration of a mixture of the corresponding dialkyltin oxide and 2-mercaptoethanol. The dimethyl derivative was obtained only as an amorphous rather insoluble solid which did not melt below 300°C, but the diethyl and dibutyl compounds were soluble, lower melting, and crystalline.

2,2-Dibutyl-1,3,2-oxathiastannolane (II, R = Bu) was chosen for investigation by single crystal X-ray diffraction. Atomic coordinates are listed in Table 1, and bond lengths and angles in Table 2, and the structure is illustrated in Fig. 1.

Individual oxathiastannolane units are intermolecularly coordinated through oxygen to give a linear polymer containing 5-coordinate tin in a distorted trigonal bipyramidal configuration. In the chain there is one short Sn–O bond distance (2.18 Å) within the stannolane ring, and one long distance (2.35 Å) between the rings. The Sn–S bond length within the ring is 2.43 Å; the Sn···S separation between monomeric units is 3.66 Å, which is within the Van der Waals distance of ca. 4.0 Å, and may represent a weak interaction.

TABLE 1

FRACTIONAL ATOMIC COORDINATES ($\times 10^4$) FOR $Bu_2\overline{SnOCH_2CH_2S}$

Atom	x	y	z	U
Sn	−2380.2(4)	0	−4893(1)	
S	−3703(2)	−1169(5)	−6009(4)	
O	−3028(5)	2282(9)	−5419(9)	
C(1)	−4249(10)	675(10)	−6412(16)	
C(3)	−2309(7)	154(20)	−2827(10)	
C(4)	−3021(10)	513(16)	−2220(11)	
C(5)	−2962(9)	268(18)	−756(11)	
C(6)	−3662(13)	1051(19)	−140(16)	
C(7)	−1318(7)	4(20)	−5995(12)	
C(8)	−1309(10)	647(16)	−7152(13)	
C(9)	−491(11)	471(19)	−7804(17)	
C(10)	−387(14)	1438(20)	−8821(19)	
C(2a)	−3965(5)	2340(13)	−5852(18)	72(2)
C(2b)	−3710(10)	2212(16)	−6540(13)	67(2)

TABLE 2

BOND LENGTHS (Å) AND ANGLES (°) FOR Bu₂$\overline{SnOCH_2CH_2S}$

|			
S–Sn	2.429(5)	O–Sn	2.180(9)
O′–Sn	2.353(9)	C(3)–Sn	2.135(12)
C(7)–Sn	2.126(12)	C(1)–S	1.773(13)
C(2a)–O	1.470	C(2b)–O	1.470
C(2a)–C(1)	1.540	C(2b)–C(1)	1.540
C(4)–C(3)	1.375(18)	C(5)–C(4)	1.522(17)
C(6)–C(5)	1.482(22)	C(8)–C(7)	1.315(19)
C(9)–C(8)	1.524(20)	C(10)–C(9)	1.350(23)
O–Sn–S	83.8(3)	O′–Sn–S	83.1(3)
O′–Sn–O	166.9(5)	C(3)–Sn–S	115.6(4)
C(3)–Sn–O	99.3(6)	C(3)–Sn–O′	86.6(6)
C(7)–Sn–S	114.5(5)	C(7)–Sn–O	103.3(6)
C(7)–Sn–O′	82.0(6)	C(7)–Sn–C(3)	126.6(6)
C(1)–S–Sn	96.8(6)	Sn″–O–Sn	133.6(2)
C(2a)–O–Sn	120.9(7)	C(2a)–O–Sn″	104.5(7)
C(2b)–O–Sn	115.2(8)	C(2b)–O–Sn″	107.5(8)
C(2a)–C(1)–S	125.4(12)	C(2b)–C(1)–S	119.0(12)
C(1)–C(2a)–O	108.0(10)	C(1)–C(2b)–O	108.0(10)
C(4)–C(3)–Sn	122.1(10)	C(5)–C(4)–C(3)	118.6(13)
C(6)–C(5)–C(4)	114.7(13)	C(8)–C(7)–Sn	126.3(11)
C(9)–C(8)–C(7)	118.3(4)	C(10–C(9)–C(8)	118.7(16)
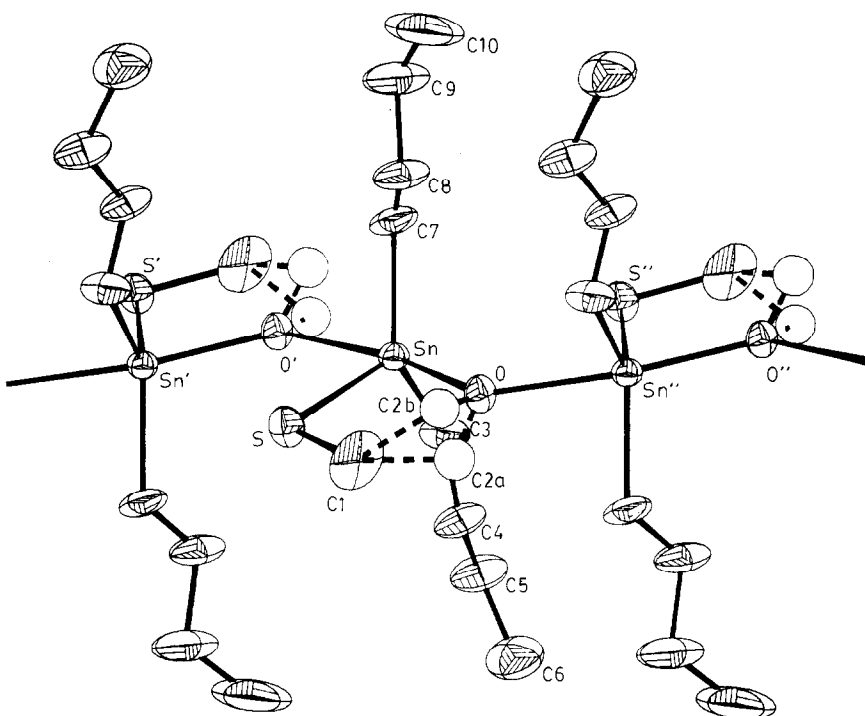
Fig. 1. The molecular structure of 2,2-dibutyl-1,3,2-oxathiastannolane. Thermal ellipsoids have been drawn at the 30% probability level. Key to symmetry operations relating designated atoms to reference atoms at (x, y, z): (′) $-0.5 - x, -0.5 + y, -1.0 - z$; (″) $-0.5 - x, 0.5 + y, -1.0 - z$.

TABLE 3. COMPARISON OF BOND LENGTHS AND ANGLES AROUND THE TIN ATOMS IN $Me_2\overline{SnSCH_2CH_2S}$, $Bu_2\overline{SnOCH_2CH_2S}$, AND $Bu_2\overline{SnOCH_2CH_2O}$

Bond length (Å) or angle (°)	$Me_2\overline{SnSCH_2CH_2S}$	$Bu_2\overline{SnOCH_2CH_2S}$	$Bu_2\overline{SnOCH_2CH_2O}$
Intramonomer			
Sn–S	2.41, 2.47	2.43	–
Intermonomer			
Sn–S	3.18	(3.66)	–
Intramonomer			
Sn–O	–	2.18	1.98, 2.10
Intermonomer			
Sn–O	–	2.35	2.50, 2.52
Intramonomer			
X–Sn–X	89.6	83.8	79.8
C–Sn–C	121.7	126.6	138.6
Intermonomer			
X–Sn–X	162.4	166.9	144.2, 145.7

The oxathiastannolane ring is in an envelope conformation, and the carbon atom attached to oxygen is disordered above or below the plane of the other four atoms of the ring, with a separation of 0.87(3) Å between the two alternative positions (atoms C(2a) and C(2b) in Fig. 1); this renders geminal butyl groups non-equivalent. The dithiastannolane ring in VI similarly exists in an envelope conformation but with the analogous carbon atom regularly displaced by about 0.9 Å from the approximate plane of the other four atoms, and the dioxastannolane ring in the δ-coordinate polymer (V) has the conformation of a twisted boat.

In some respects, the structure of the oxathiastannolane is intermediate between that of the dioxastannolane V (R = Bu) and the dithiastannolane VI, though closer to the latter. Some important bond lengths and angles are compared in Table 3, and illustrated for the oxathiastannolane (R = Bu^t) and dithiastannolane III (R = Me) in IX and X. The significance of the comparison is weakened however by the fact that the alkyl group R is different in the two compounds, and as is discussed below,
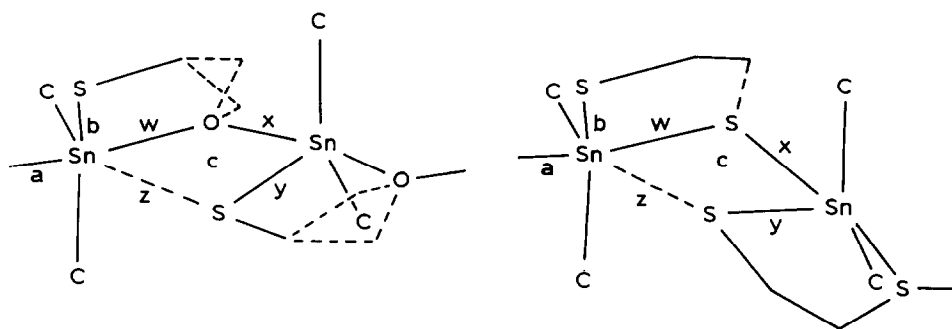
(IX)

Angle(°)	Length (Å)
a 126.6	w 2.18
b 83.8	x 2.35
c 133.6	y 2.43
	z 3.66

(X)

Angle (°)	Length (Å)
a 121.7	w 2.47
b 89.6	x ʹ3.18
c 113.5	y 2.41
	z 4.70

TABLE 4

MELTING POINTS AND MÖSSBAUER AND NMR SPECTRA OF OXATHIASTANNOLANES

	$Me_2\overline{SnOCH_2CH_2S}$	$Et_2\overline{SnOCH_2CH_2S}$	$Bu_2\overline{SnOCH_2CH_2S}$
M.p. (°)	> 300	131–133	93
Mössbauer spectra δ	1.23	1.41	1.38
(mm s⁻¹) ΔE_q	2.72	2.95	2.92
$\delta(^1H)$ (CDCl₃)	2.84(SCH₂) [a]	2.79(SCH₂) [d]	2.77(SCH₂) [f]
	3.60(OCH₂) [b]	3.63(OCH₂) [e]	3.63(OCH₂) [g]
	0.73(CH₃) [c]	ca. 1.18(C₂H₅)	
$\delta(^{13}C)$ (CDCl₃)	31.6(SCH₂)	31.5(SCH₂)	31.3(SCH₂)
	62.1(OCH₂)	63.3(OCH₂)	62.4(OCH₂)
	3.5(CH₃)	15.0(CH₂)	22.9($C_\alpha H_2$) [h,i]
		9.8(CH₃)	27.5($C_\beta H_2$) [j]
			26.6($C_\delta H_2$) [k]
			13.4(CH₃)
$\delta(^{13}C)$ (solid)	32.8 32.4(SCH₂)	33.3(SCH₂)	32.4(SCH₂)
	65.6(OCH₂)	67.4(OCH₂)	66.0(OCH₂)
		16.8(CH₂)	24.3, 23.6($C_\alpha H_2$)
	5.8(CH₃) [l]	10.7(CH₃)	29.3, 28.6 ⎫
			29.4,? ⎬ $(CH_2(\beta + \gamma))$
			15.2, 15.0(CH₃)
$\delta(^{119}Sn)$ (CDCl₃)	−21	−34	−34
$\delta(^{119}Sn)$ (solid)	−51	−31	−41

$J(^{117/119}Sn^{-1}H)$ (Hz): [a] 18, [b] 44, [c] 68, [d] < 24, [e] 43, [f] ca. 16, [g] 44, $J(^{119}Sn^{-13}C)$ 600±6, [j] 34, [k] 85, [l] 606. [h] $J(^{119}Sn^{-13}C)$ 528 Hz. [i] $J(^{117}Sn^{-13}C)$ 503 Hz.

the structures of the dimethyl- and the dibutyl-oxathiastannolanes may be significantly different.

Mössbauer and NMR data for the three oxathiastannolanes are given in Table 4 and can be discussed in the light of these X-ray diffraction results.

Hager et al. [13] carried out point charge calculations for various possible structures of dialkyloxathiastannolanes, and obtained the values of the quadrupole coupling constants which are shown for the structures XI, XII, and XIII.

(XI)   (XII)   (XIII)

−2.305 mm s⁻¹   +2.143 mm s⁻¹   −2.514 mm s⁻¹
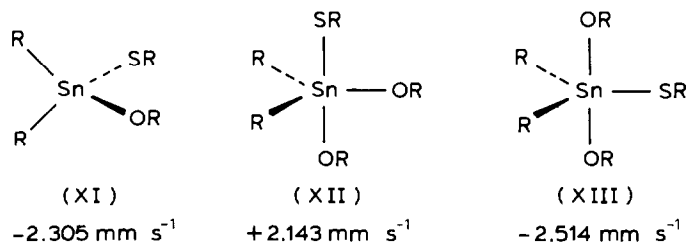
A study of the temperature dependence of the recoil-free fraction of the dimethyl compound suggested that the compound was polymeric and this, together with the observed quadrupole splitting of 2.72 mm s⁻¹ was taken to favour structure VIII, similar to that which we have now established for the dibutyl derivative. We note however that the values of δ and of ΔE_q for the dimethyl compound is significantly different from that of the diethyl and dibutyl compounds.
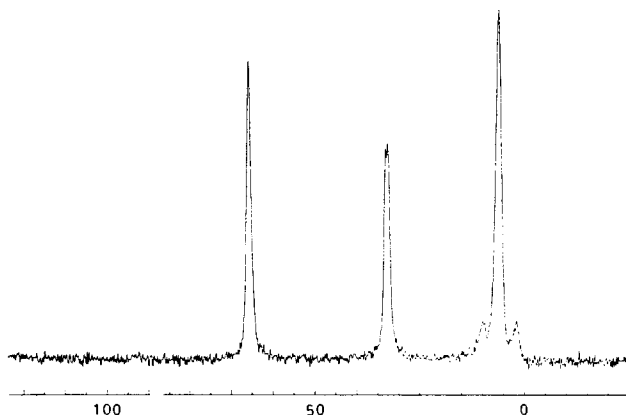
Fig. 2. CP/MAS solid state ^{13}C NMR spectrum of 2,2-dimethyl-1,3,2-oxathiastannolane.

Sham and Bancroft [19] have used the expression in eq. 1 to relate the quadrupole coupling to θ which is $(180° -$ angle R–Sn–R)/2; s is the partial quadrupole coupling of the alkyl groups R and is taken to be -1.03 mm s^{-1}.

$$\Delta E_q = 4(s)(1 - 3 \sin^2\theta \cos^2\theta)^{1/2} \tag{1}$$

Application of this formula to dibutyloxathiastannolane predicts that the angle R–Sn–R should be 126° in agreement with the value of 126.6° which we find, and for the dimethyl and diethyl compounds it predicts the angles of 120 and 126°, respectively.

Lockhart has used the eq. 2 in the same sense for relating $^1J(^{13}C-^{119}Sn)$ (Hz) in di- and tri-methyltin compounds to the C–Sn–C angle ϕ [20].

$$|^1J(C-Sn)| = 10.7\phi - 778 \tag{2}$$

The $^{117/119}$Sn satellites were not observed for the dimethyloxathiastannolane in solution because of its low solubility, but in the solid state (Fig. 2) the satellites were apparent with $^1J(C-Sn)$ 600 ± 6 Hz, which by Lockhart's equation would correspond to a C–Sn–C angle of 128°.

The ^{13}C NMR spectra of the dimethyl and diethyl derivatives in solution or in the solid state (Figs. 2 and 3) each show the presence of only one type of alkyl group, but the dibutyl derivative is interesting in that it shows only one type of butyl group in solution, but two in the solid state (Fig. 4), the separation between corresponding signals being greatest for the C_α and least for the C_δ of the butyl groups. Similarly 2,2-dimethyl-1,3,2-dithiastannolane shows one type of methyl group in solution, but two in the solid state (Fig. 5). Clearly a variable temperature solid state NMR study of these compounds might give very interesting results.

Holeček and Lyčka [21] have recently proposed the modification of Lockhart's equation, as shown in eq. 3, for correlating $^1J(C-Sn)$ and ϕ in di-n-butyltin(IV) compounds.

$$|^1J(C-Sn)| = (9.99 \pm 0.73)\phi - (746 \pm 100) \tag{3}$$

By this equation, our observed value of $^1J(C-Sn)$ in solution of 528 Hz would correspond to an angle of about 128°, to be compared with the value of 126.6° which we find by X-ray crystallography.
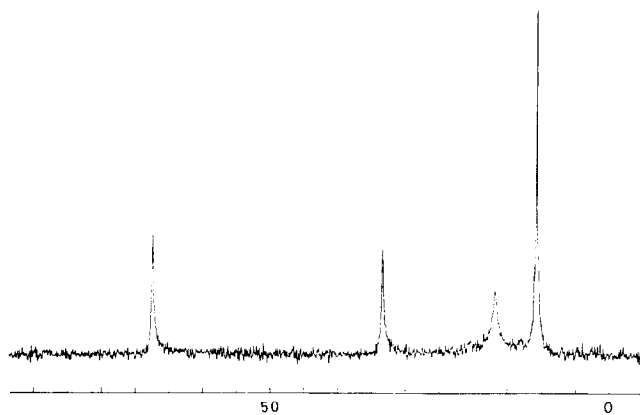
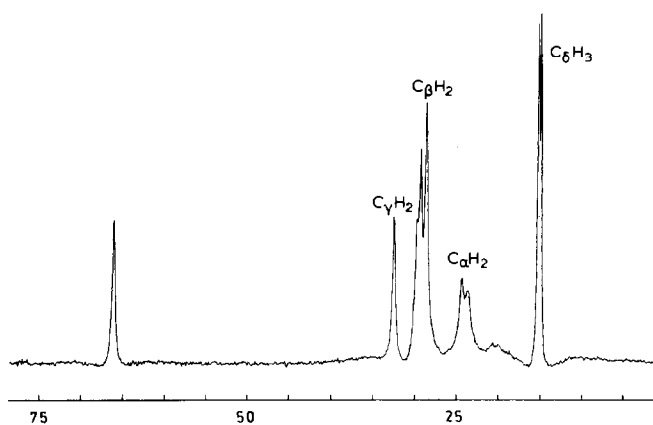Fig. 3. CP/MAS solid state ^{13}C NMR spectrum of 2,2-diethyl-1,3,2-oxathiastannolane.

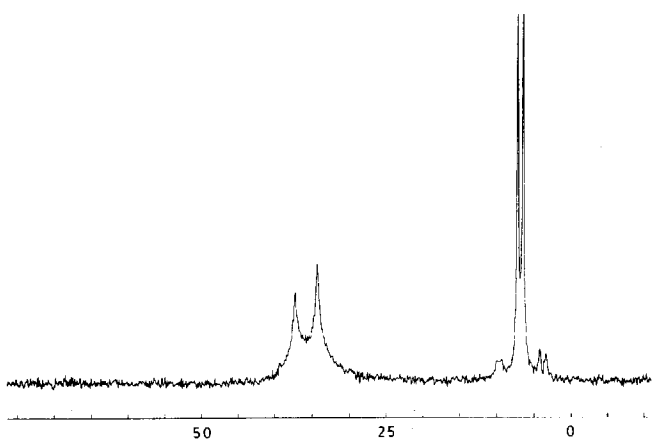Fig. 4. CP/MAS solid state ^{13}C NMR spectrum of 2,2-dibutyl-1,3,2-oxathiastannolane.

Fig. 5. CP/MAS solid state ^{13}C NMR spectrum of 2,2-dimethyl-1,3,2-dithiastannolane. δ 6.3 and 7.2 (CH$_3$; ^1J(Sn–C) 445 Hz), 34.2 and 37.2 (CH$_2$S).
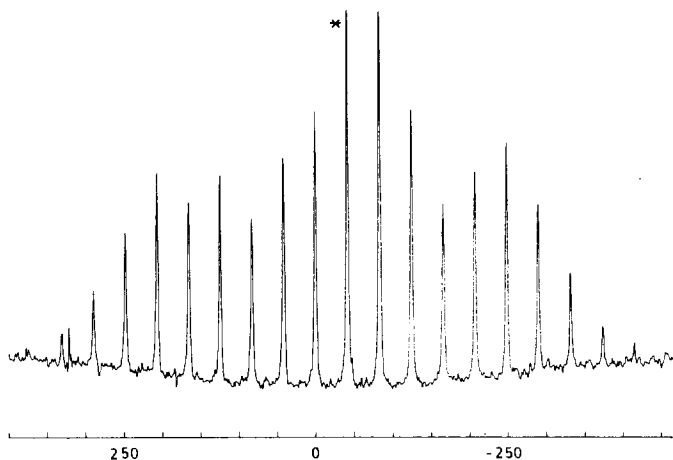
Fig. 6. CP/MAS solid state ^{119}Sn NMR spectrum of 2,2-dibutyl-1,3,2-oxathiastannolane.

The ^{119}Sn NMR spectrum of dibutyloxathiastannolane in the solid state is illustrated in Fig. 6. The ^{119}Sn NMR chemical shifts of the diethyl and dibutyl oxathiastannolanes in the solid state are close to those in solution, but in the case of the dimethyl compound there is a substantial shift in the direction of increased coordination.

The intensity and separation of satellites due to [117]Sn/[119]Sn coupling in both solution and the solid state provides a valuable method for investigating the aggregation of organotin compounds, but we were unable to detect these satellites for any of the three oxathiastannolanes. In solution, the [119]Sn signal for the dibutyl derivative had a width of 200 Hz at the base, and any coupling smaller than this would be lost in the line width.

The differences in melting point, solubility, Mössbauer spectra, and change in ^{119}Sn NMR chemical shift between solution and solid that we find for the dimethyl derivative, and the diethyl and dibutyl derivatives, appear to imply that in the solid state the dimethyl compound is more strongly associated than the dibutyl compound. Although all three compounds probably have the same basic structure of a linear polymer, in the dimethyl compound to O–Sn bonds between the units in the chain may be stronger, or perhaps the weak S–Sn interaction that we pointed to in the butyl derivative may play a larger part in the bonding of the methyl compound. Unfortunately the non-crystalline nature of the dimethyl compound has precluded a study by X-ray diffraction.

Experimental

Preparations

The oxathiastannolanes were prepared by azeotropic dehydration of a mixture of 2-mercaptoethanol and the appropriate dialkyltin oxide in benzene. The sample of dibutyloxathiastannolane for X-ray diffraction studies was recrystallised from methanol.

Mössbauer spectra

Mössbauer spectra were recorded at the International Tin Research Institute. The samples were cooled in liquid nitrogen, and isomer shifts are quoted against SnO_2 as standard.

NMR spectra

Solution spectra were recorded on a Varian XL200 spectrometer. Solid state spectra were recorded at Royal Holloway and Bedford New College on the U.L.I.R.S. Bruker MSL300 instrument. Typical conditions for running the ^{119}Sn spectra were as follows: operating frequency 111.914 MHz, rotation rate ca. 4500 Hz, number of scans ca. 200, recycle delay time 5 s, contact time 10 ms. Chemical shifts are quoted with $\delta(Me_4Sn) = 0$, and the magnetic field was calibrated against $(Me_3Sn)_4C$, δ +48.2. We are grateful to Dr. T.N. Mitchell (Dortmund) for a sample of this compound.

X-ray crystallography

Crystal data. $C_{10}H_{22}OSSn$, $M = 309.0$, monoclinic, a 15.557(2), b 8.292(3), c 10.367(2) Å, β 97.42(2)°, U 1325 Å^3, space group $C2$, $Z = 4$, D_c 1.548 g cm^{-3}, $F(000) = 624$, λ 0.71069 Å, $\mu(Mo\text{-}K_\alpha)$ 20.5 cm^{-1}, crystal size $0.13 \times 0.10 \times 0.45$ mm.

Data collection. Unit cell parameters and intensity data were obtained by following previously detailed procedures [22] using a CAD4 diffractometer operating in the ω–2θ scan mode, with graphite monochromated Mo-K_α radiation. A total of 1259 unique reflections were collected. The segment of reciprocal space scanned was: (h) $-18\text{-}>18$, (k) $0\text{-}>9$, (l) $0\text{-}>12$. The reflection intensities were corrected for absorption, using the azimuthal-scan method [23]; maximum transmission factor 0.99, minimum value 0.96.

Structure solution and refinement. The structure was solved by the application of routine heavy-metal methods, and refined by full-matrix least squares (SHELX76 [24]). One of the carbon atoms of the oxathiastannolane ring, the one attached to oxygen, is disordered. Thus, two positions were refined for this atom (C(2a) and C(2b) in Fig. 1), each with an occupancy factor of 0.5. During refinement, bond lengths of these positions were constrained (instruction DFIX in SHELX76, C(2a, b)–O 1.470, C(2a, b)–C(1) 1.540 Å).

All non-hydrogen atoms, except the disordered carbon atom, were refined with anisotropic thermal coefficients. The final cycle of refinement included all hydrogen atoms of the n-butyl groups in calculated positions (C–H 1.08 Å, U 0.10 Å^2). Hydrogen atoms of the oxathiastannolane ring were not included in the refinement. The final values of R and R_w were 0.043 and 0.048 respectively for the 122 variables and 1013 data for which $|F^2| > 3\sigma(F^2)$.

The function minimised was $\Sigma_w(|F_o| - |F_c|)^2$ with the weight, w, being defined as $1/[\sigma^2(F) + 0.013F^2]$. The absolute configuration was shown to be correct (instruction UNDO-1 in SHELX76, $R = 0.045$). A final difference map had peaks of up to 0.7 e Å^{-3} near the Sn atom but was elsewhere featureless.

Atomic scattering factors and anomalous scattering parameters were taken from refs. 25 and 26, respectively.

All computations were made on a DEC VAX-11/750 computer.

Tables of isotropic hydrogen atom coordinates, anisotropic thermal coefficients,

and F_o/F_c values have been deposited as supplemenary data, and are available on request from the Director of the Cambridge Crystallographic Data Centre, University Chemical Laboratory, Lensfield Road, Cambridge CB2 1EW (U.K.). Any request should be accompanied by a full literature citation for this communication.

Acknowledgements

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