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The structures of 2,2-dialkyl-1,3,2-dithiastannolanes

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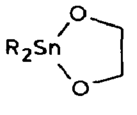
Abstract

The structure of 2,2-dibutyl-1,3,2-dithiastannolane in the solid state has been determined by single crystal X-ray diffraction. Individual molecules interact weakly with two neighbouring molecules to form two long (3.688 Å) Sn–S coordinate bonds. The tin atoms can therefore be regarded as being weakly 6-coordinate. In contrast, in the crystal, 2,2-dimethyl-1,3,2-dithiastannolane forms one short coordinate bond (3.18 Å) to a neighbouring molecule, and the tin can be regarded as being strongly 5-coordinate.

The ^{13}C and $^{117/119}$Sn NMR spectra of the dimethyl-, diethyl-, diisopropyl-, and dibutyl-stannolanes, and of spirobis(ethane-1,2-dithiolato)tin have been recorded in solution and the solid state, and correlated with the evidence from X-ray crystallography. It is argued that the diethyl- and diisopropyl-stannolanes have structures similar to that of the dibutyl derivative.

Introduction

The dioxa-, oxathia-, and dithia-stannolanes (I, II, and III respectively) provide an important series of compounds for studying the relation between the molecular structures and the nature of the ligands. All three types of compound form 5- and 6-coordinate complexes more readily than do their acyclic analogues, perhaps because the change from trigonal to bipyramidal or octahedral geometry relieves strain in the 5-membered ring. In the absence of an external ligand, self-association may occur, to a degree which depends inter alia on the nature of the internal ligand (O or S), the physical state (solution or solid), and the identity of the alkyl groups and of the diol moiety.



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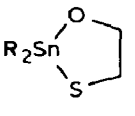

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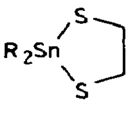

(III)

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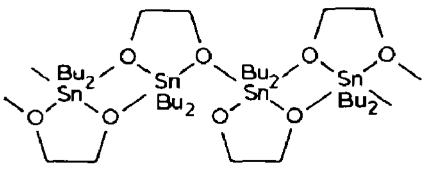
We have discussed the structures of the dioxa- [1] and the oxathia-stannolanes [2] in previous papers.

The dioxastannolanes (I) are usually dimeric in solution, and this dimerisation can result in regiospecific reaction of the stannolanes derived from unsymmetrical diols such as carbohydrates [3]. In the crystal, the degree of association may increase to a degree which depends on the bulk of the diol moiety: 2,2-dibutyl-1,3,2-dioxastannolane itself is an infinite 6-coordinate polymer (IV) [1], but the dibutyltin derivative of methyl 4,5-di-*O*-benzylidene-α-D-mannopyranoside is a pentamer [4], and the corresponding glucose derivative is a dimer [5].



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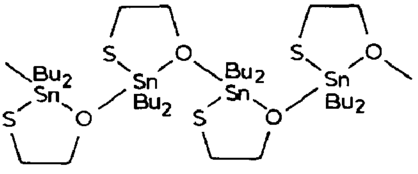
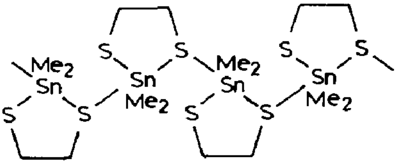


(V)

The oxathiastannolanes (II) are also usually dimers in solution. In the crystal, 2,2-dibutyl-1,3,2-oxathiastannolane is an infinite 5-coordinate polymer associated through oxygen as shown in V, but the intermonomer Sn–S separation of 3.66 Å is within the Van der Waals distance of ca. 4.0 Å, and may indicate a further weak interaction towards 6-coordination [2]. The high melting point and lower solubility of 2,2-dimethyl-1,3,2-oxathiastannolane suggest that in this compound this tendency towards 6-coordination may be stronger.

Sulphur thus appears to be a weaker ligand than oxygen. This is bourne out in the dithiastannolanes (III), which are 4-coordinate monomers in solution [6–9]. In the crystal, 2,2-dimethyl-1,3,2-dithiastannolane forms a linear 5-coordinate polymer (VI) containing tin in a distorted trigonal bipyramidal environment [10,11].



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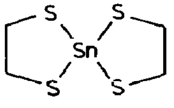


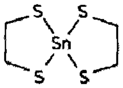
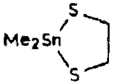
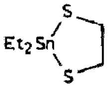
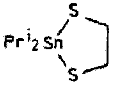
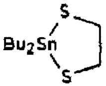
(VII)

The related spirobis(ethanedithiolato)tin compound (VII) in the crystal shows two slightly different Sn–S bond lengths within the rings (2.405(1) and 2.388(1) Å), and two intermolecular Sn–S bond distances of 3.764 and 3.811 Å, which are less than the Van der Waals distance [12].

We report here a study of the structures of the dialkyldithiastannolanes (III: R = Me, Et, Pr^i, and Bu) and of the spirobisdithiastannolane (VII), making particular use of the techniques of $^{117/119}Sn$ NMR spectroscopy in solution and in the solid state. We have also determined the structure of dibutyl-1,3,2-dithiastannolane by single-crystal X-ray diffraction.

Table 1

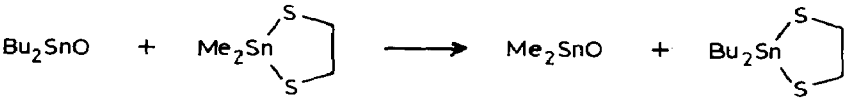
NMR and Mössbauer spectra of dithiastannolanes

		NMR spectra												Mössbauer spectra	
		$\delta(^{1}H)$ Solution		$\delta(^{13}C)$		$\delta(^{117/119}Sn)$		$\delta(Sn)$ tensor components						δ	ΔEq
				Solution	Solid	Solution ^{a}	Solid ^{b}	σ_{11}	σ_{22}	σ_{33}	δ	Anis	η		
	CH_2S	3.23	CH_2S	35.6	37.1, 37.4	277	241	145.8	264.0	314.4	73	109	1.62	1.40	1.03
	CH_2S CH_3Sn	3.00 0.78 ^{c}	CH_2S CH_3Sn	36.2 1.2 ^{d}	34.2, 37.2 6.34, 7.06 ^{e}	193	66.9	−84.5	28.1	257.1	190	285	0.59	1.50	2.60
	CH_2S C_2H_5Sn	3.2 1.4–1.8	CH_2S CH_2 CH_3	36.3 10.23 11.70	38.1 17.4 11.6	199	188	38.7	249.3	276.4	88	132	2.38	1.56	2.34
	CH_2S CH CH_3	3.00 ^{f} 2.02 (sept) 1.37 (d ^{g}) ^{h}				191	179	81.2	199.7	257.0	78	116	1.52	1.64	2.15
	CH_2S C_4H_9Sn	3.00 0.9–1.8	CH_2S $CH_2\alpha$ $CH_2\beta$ $CH_2\gamma$ CH_3	36.3 20.0 ^{i} 28.15 ^{j} 27.6 13.58	37.2 (br) 23.6 (br) 29.0 15.5	193	175	120.1	194.9	196.4	26	39	2.89	1.53	2.34

[a] All on ^{119}Sn. [b] All except for the bis(ethanedithiolato) compound on ^{117}Sn. [c] $^{2}J(Sn–H)$ 62 Hz. [d] $^{1}J(Sn–C)$ 372 Hz. [e] $^{1}J(Sn–C)$ 445 Hz. [f] $^{3}J(Sn–H)$ 33 Hz. [g] $^{3}J(H–H)$ 8 Hz. [h] $^{2}J(^{117/119}Sn–H)$ 96.2/100.4 Hz. [i] $^{1}J(Sn–C)$ 342 Hz. [j] $^{2}J(Sn–C)$ 13 Hz.

Results

Dimethyl-1,3,2-dithiastannolane (III, R = Me) and spirobis(1,2-ethanedithiolato)-tin (VII) were prepared by treating dimethyltin dichloride or tin tetrachloride respectively with the dipotassium salt of ethane-1,2-dithiol. The diethyl- and diisopropyl-dithiastannolanes (III, R = Et, Pr^i) were obtained from the corresponding dialkyltin dichlorides, ethanedithiol, and pyridine, and the dibutyl compound (III, R = Bu) by the novel method of heating dibutyltin oxide with dimethyldithiastannolane (eq. 1).



(1)

Table 1 lists the ^{1}H, ^{13}C, and ^{119}Sn NMR data for the compounds in solution, and the ^{13}C and ^{119}Sn (or ^{117}Sn) * data obtained by magic angle spinning with cross polarisation (CPMAS) in the solid state. Mössbauer isomer shifts and quadrupole couplings are also included.

2,2-Dibutyl-1,3,2-dithiastannolane was chosen for structural determination by single crystal X-ray diffraction, to provide a comparison with the known structures

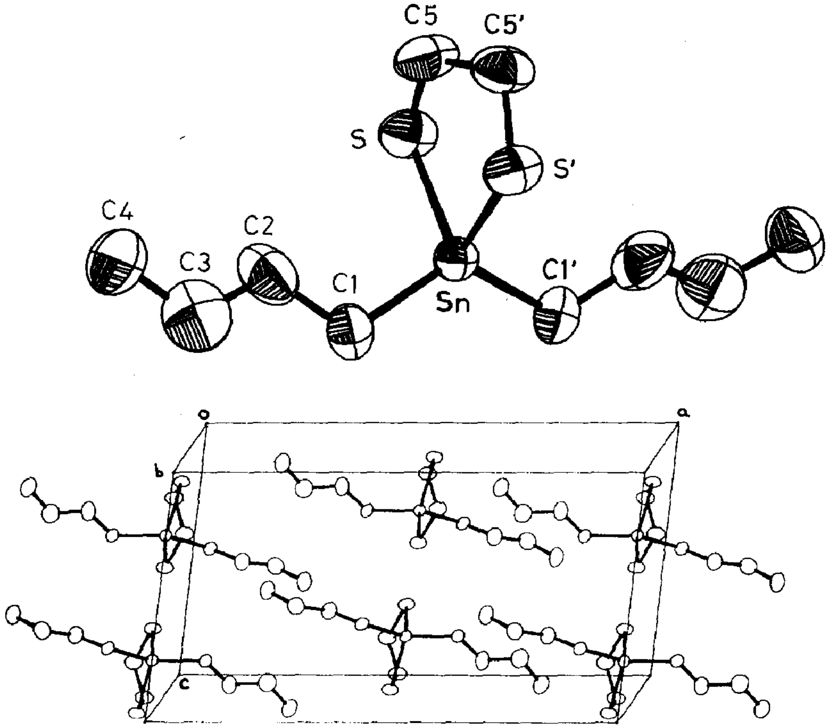


Fig. 1. Molecular and crystal structure of 2,2-dibutyl-1,3,2-dithiastannolane.

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* For operational reasons (see Experimental), we have found it better to record solid state ^{117}Sn rather than ^{119}Sn NMR spectra. Values of the chemical shift should be same from both nuclii.

Table 2

Fractional Atomic Coordinates for 2,2-Dibutyl-1,3,2-Dithiastannolane [a]

Atom	x	y	z	$B(\text{Å}^2)$
Sn	0.000	−0.00070(7)	0.250	3.93(1)
S	−0.0072(1)	0.2147(2)	0.4197(1)	5.50(4)
C(1)	0.1041(4)	−0.1296(9)	0.2745(6)	5.4(2)
C(2)	0.1681(6)	−0.0173(9)	0.349(1)	7.6(2)
C(3)	0.2437(6)	−0.086(1)	0.354(1)	9.1(3)
C(4)	0.3018(6)	0.017(1)	0.437(1)	8.5(3)
C(5)	0.0152(6)	0.3933(8)	0.3208(6)	6.2(2)

[a] The $B_{eq.}$ values are calculated according to the equation $B_{eq.} = \frac{4}{3}\Sigma_i\Sigma_j B_{ij} a_i a_j$.

Table 3

Bond lengths (Å) and angles (°) for 2,2-dibutyl-1,3,2-dithiastannolane

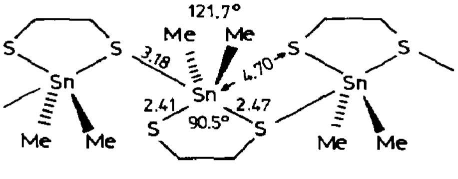
Sn–S	2.414(2)		
Sn–C(1)	2.115(8)		
S–C(5)	1.796(7)		
C(1)–C(2)	1.560(12)		
C(2)–C(3)	1.457(15)		
C(3)–C(4)	1.484(15)		
C(5)–C(5′)	1.444(12)		
Sn–S [a,b]	3.688(3)		
C(1)–Sn–S	112.7	S–C(5)–C(5′)	117.0
C(1)–Sn–C(1) [c]	122.6	Sn–C(1)–C(2)	111.6
C(1)–Sn–S′	106.9	C(1)–C(2)–C(3)	115.4
S–Sn–S′	90.5	C(2)–C(3)–C(4)	113.5
Sn–S–C(5)	97.5	S–Sn–S [a]	72.2
		S–Sn–S [b]	161.7

[a] Symmetry operators $\bar{x}$, $\bar{y}$, $1-z$. [b] x, $\bar{y}$, $-\frac{1}{2}+z$. [c] $\bar{x}$, y, $\frac{1}{2}-z$.

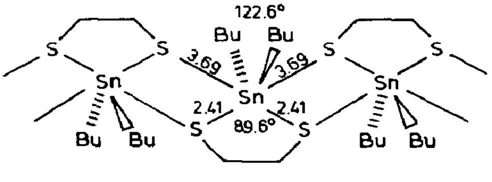
of dibutyldioxa- and dibutyloxathia-stannolanes. Atomic coordinates are listed in Table 2, and bond lengths and bond angles in Table 3, and the molecular and crystal structures are illustrated in Fig. 1.

Discussion

We will first discuss the structure of dibutyldithiastannolane as shown by single crystal X-ray diffraction, and then analyse the NMR data.



(VIII)



(IX)

X-Ray crystallography

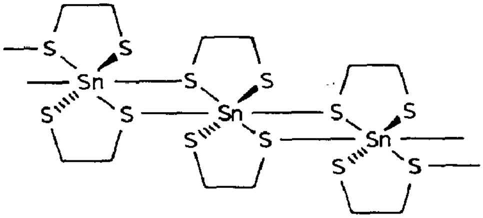
The principal features of the structures of dimethyl- and dibutyl-dithiastannolane are compared in formulae VIII and IX, where bond lengths are given in Å.

Individual molecules in both have very similar structures, but the nature of the interaction of these units in the crystal is significantly different.

In VIII one of the two sulphur atoms interacts strongly with a neighbouring tin atom to give a linear polymer with a coordinate bond length of 3.18 Å. The second sulphur atom is 4.70 Å away from neighbouring tin and is not involved in bonding between the monomeric units. The dithiolato ligand in VIII is thus rendered anisobidentate, with Sn–S bond lengths within the ring of 2.41 Å and 2.47 Å [10,11].

On the other hand, in IX, each tin atom is 3.69 Å distant from a sulphur atom on each of two neighbouring dithiastannolane units forming an S...Sn...S angle of 125.5 ° C. This is significantly greater than a normal S → Sn coordinate bond length (ca. 2.45 Å), but less than the sum of the Van der Waals radii (ca. 4.0 Å). Thus whereas the tin atom in VIII has unambiguously 5 ligands in its coordination sphere, the dibutyl analogue X can be regarded as having six, though two are only weakly interacting.

In the crystal, the spirobis(ethanedithiolate) (VII) has a structure closer to that of IX than of VIII. The rings are only slightly anisobidentate, with Sn–S bond lengths of 2.405(1) and 2.388(1) Å, and the tin interacts weakly with a sulphur atom from each of two neighbouring units, with S → Sn separations of 3.764 Å and 3.811 Å, as illustrated in X [12].



(X)

NMR spectroscopy

In solution, the dialkyldithiastannolanes are monomolecular, and the half-chair rings are rapidly fluxional on the NMR time scale [9]. Towards the NMR probe, the molecules therefore show apparent C_{2v} symmetry: the ^{1}H and ^{13}C NMR spectra show a single set of signals for the two equivalent alkyl groups, and a single signal for the two methylene groups in the dithiastannolane ring.

Any structural changes which occur when the compounds solidify will show up in the ^{13}C NMR spectra by changes in the chemical shifts, and sometimes, if local C_{2v} symmetry is lost, by splitting of the single sets of ^{13}C signals into duplicate sets. Further, any increase in the degree of association involving tin will show itself by an upfield shift of δ ($^{117/119}Sn$) *.

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* When the structures are the same in solution and the solid state, as they are for example in ($Bu^{t}_2SnO_3$) [13] and $Bu^{t}_2Sn(SCH_2)_2$ [14], the NMR chemical shifts usually do not change more than they would between two different non-interactive solvents.

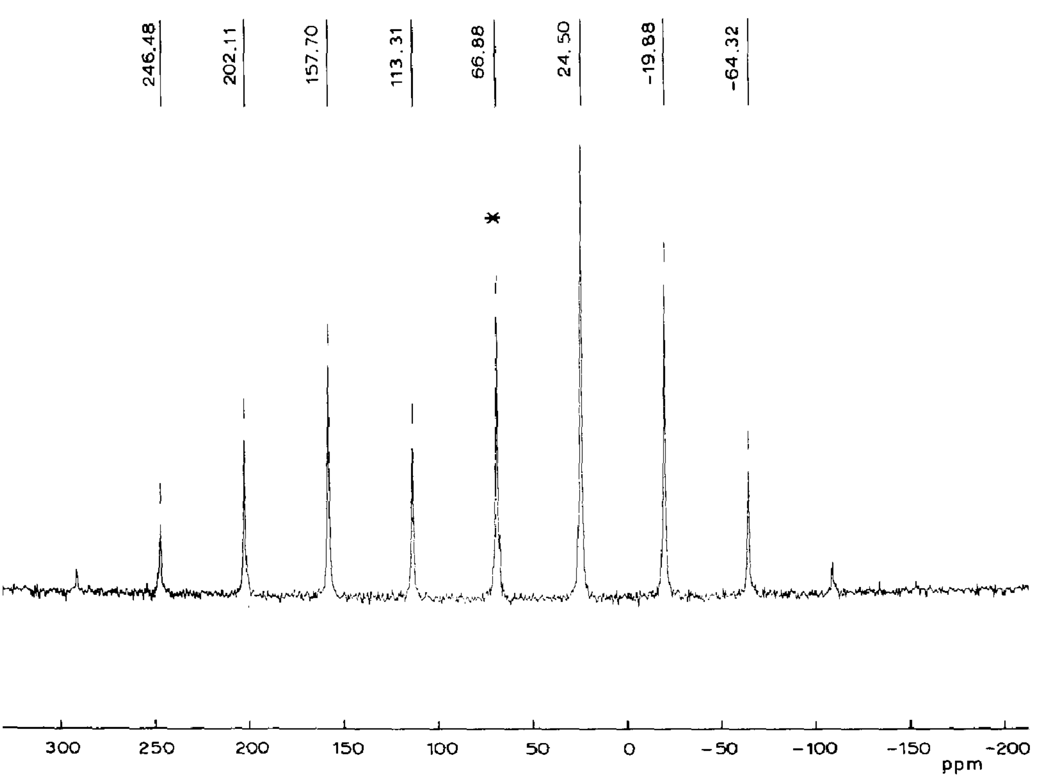

Fig. 2. CP/MAS solid state ^{117}Sn NMR spectrum of 2,2-dimethyl-1,3,2-dithiastannolane.

Thus when monomeric dimethyldithiastannolane (III, R = Me) crystallises from solution to give the 5-coordinate polymer VIII, the single ^{13}C signal for the CH_2S group at δ 36.2 splits into two signals at δ 34.2 and 37.2 indicating loss of C_{2v} symmetry. The spectrum is illustrated in ref. 2. The two methyl groups are also rendered non-equivalent in the crystal, and the single signal at δ 1.2 (${}^1J({}^{117/119}Sn–C)$ 372 Hz) splits into a pair of signals at δ 6.34 and 7.06 (${}^1J({}^{117/119}Sn–C)$ 445 Hz) *.

In particular, the value of δ (${}^{117/119}$Sn) 193 in solution shows a large upfield shift to δ 66.9 when the 5-coordinate polymer is formed (Fig. 2).

The dibutyldithiastannolane (III, R = Bu) behaves differently. Solidification is accompanied by a downfield shift of the ^{13}C signals of the butyl and CH_2S groups, but the signals remain as singlets although those for the α-CH_2 and CH_2S groups broaden (Fig. 3). At the same time δ (${}^{117/119}$Sn) moves upfield by 18 ppm and the static band shape (Fig. 4) as defined by the tensor components σ_{11}, σ_{22}, and σ_{33}, is substantially different for that for III (R = Me) (Fig. 2) **.

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* Lockhart, Manders, and Zuckerman [15] have proposed the following empirical expression for relating the Me–Sn–Me angle θ to the ^{119}Sn–^{13}C hyperfine coupling

$$|{}^1J({}^{119}Sn–{}^{13}C)| = 10.7\phi - 778$$

Our observed values of ${}^1J$ would lead by this expression to the predicted values of ϕ 113.7° in solution, and 123.4° in the solid state, to be compared with the value of 121.7° determined crystallographically.

** Holeček and Lyčka [16] have proposed the equation

$$|J^1({}^{119}Sn–C)| = (9.99 \pm 0.73)\phi - (746 \pm 100)$$

correlating spin–spin coupling and the C–Sn–C angle (ϕ) in dibutyltin(IV) compounds. This would suggest an angle θ of 110° ±18° for III (R = Bu) in $CHCl_3$.

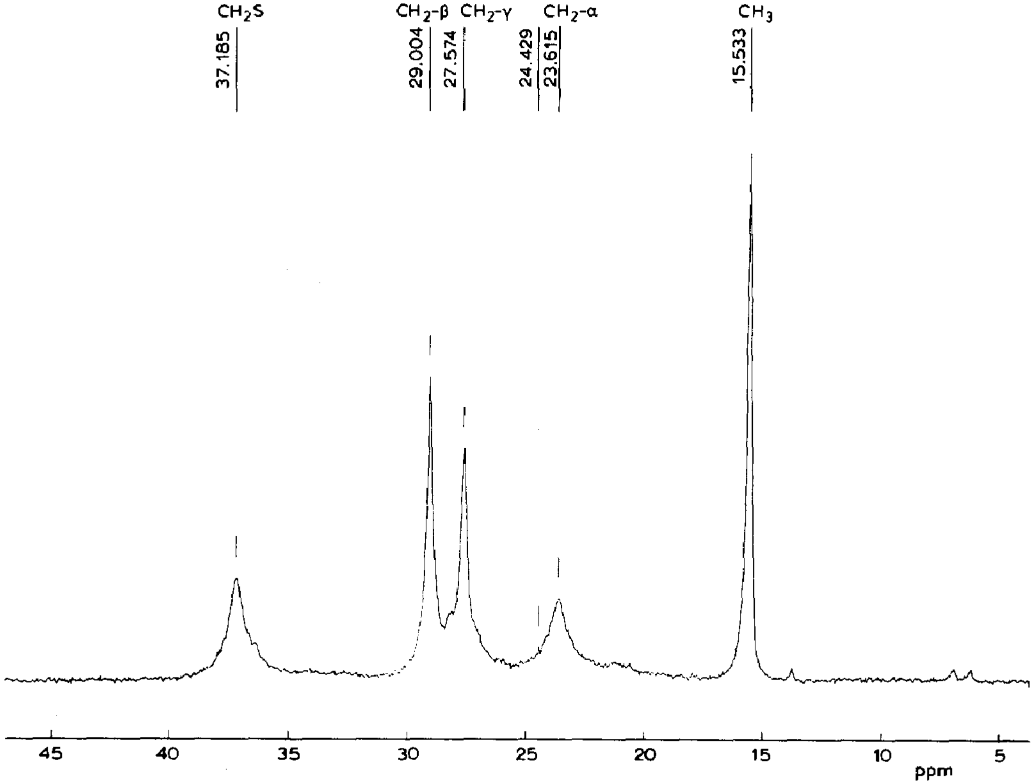

Fig. 3. CP/MAS solid state ^{13}C NMR spectrum of 2,2-dibutyl-1,3,2-dithiastannolane.

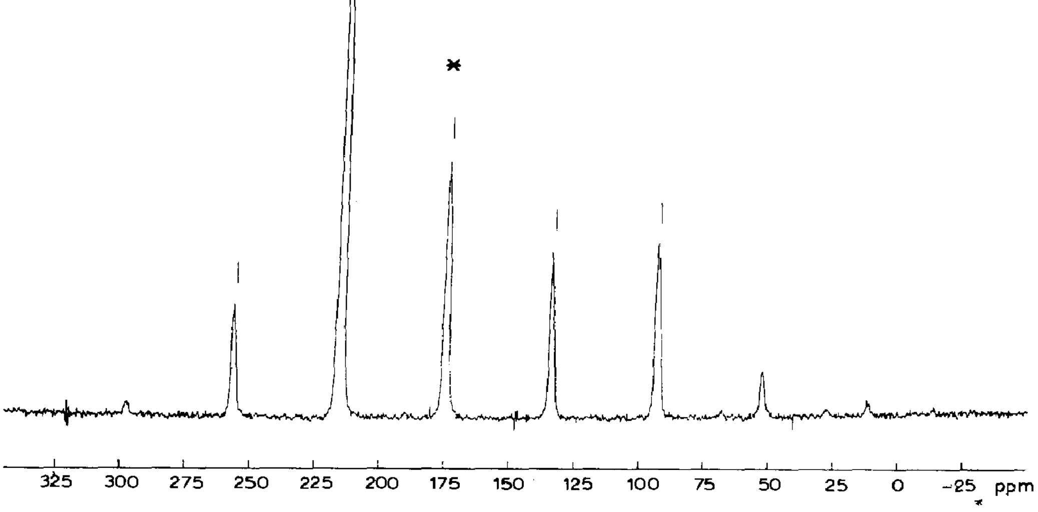

Fig. 4. CP/MAS solid state ^{119}Sn NMR spectrum of 2,2-dibutyl-1,3,2-dithiastannolane.

This is in accord with the different structure of III (R = Bu), as determined crystallographically (Fig. 1), in which the tin atom forms two weak coordinate bonds with sulphur from two neighbouring dithiastannolane units, but the individual molecules preserve their C_{2v}-axis of symmetry. The broadening of the α-CH_2 and CH_2S signals may be associated with inversion within the half-chair dithiastannolane rings as identified by Abel et al. [9].

The diethyl- and diisopropyl-dithiastannolanes (III, R = Et and ^iPr) show NMR characteristics resembling those of III (R = Bu) rather than III (R = Me).

The NMR spectra of the spirobis(ethanedithiolate) (VII) can be understood on a similar basis. Weak intermolecular coordination by two of the four sulphur atoms separates the methylene groups into non-equivalent pairs which are partially resolved in the solid state ^{13}C NMR spectrum, and at the same time the tin resonance shifts upfield by 36 ppm.

Mössbauer spectra

Sham and Bancroft [17] have used the expression in eq. 2 to relate the quadrupole coupling to θ which is (180° − angle RSnR)/2, and s is the partial quadrupole coupling of the alkyl groups R, which is taken to be -1.03 mm s^{-1}.

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

Our observed quadrupole couplings would by this equation predict R–Sn–R angles of 116° when R = Me, 108° when R = Et or Bu, and 100° when R = Pr^i. These predicted angles are to be compared with the values of 121.7° when R = Me, and 122.6° when R = Bu, as determined crystallographically.

The Mössbauer spectrum of the bis(ethanedithiolate) has been thoroughly discussed by Herber and Leahy [18]. The observation of a quadrupole-split doublet implies that the tin is not simply tetrahedrally four-coordinate, and the temperature dependence of the recoil-free fraction implies the association of the molecules into one-dimensional chains as confirmed by X-ray diffraction, as illustrated in X.

Conclusion

These studies have emphasised that, once crystal structures are known for suitable calibrant compounds, high resolution solid state NMR spectroscopy provides a rapid and powerful method for determining the structures of organotin compounds in the solid state. In due course, by an analysis of the tensor components of the chemical shift, it should be possible to make the interpretation more quantitative.

A second point which is underlined by these studies is the difference in structure and other properties which is often observed between methyl and higher alkyl derivatives of tin(IV) (and indeed between methyl and higher alkyl derivatives of other metals). For example trimethyltin hydroxide is very resistant to dehydration, whereas the higher homologues dehydrate readily. Similarly the higher melting point, and low solubility, and the solution and solid state NMR parameters of 2,2-dimethyl-1,3,2-oxathiastannolane imply that it is more firmly associated than are the higher homologues [2]. In the present study we have shown equimolar amounts of dibutyltin oxide and 2,2-dimethyl-1,3,2-dithiastannolane react to give dimethyltin oxide and the dibutylstannolane in good yield, and our X-ray and NMR data show that whereas the dimethylstannolane is firmly 5-coordinate in the solid state, the dibutyl, and in all probability the diethyl and diisopropyl homologues, are weakly 6-coordinate.

Although many of these special properties of methyltin compounds may be seen as examples of the steric protection by larger alkyl groups against intermolecular association, it seems likely that an electronic factor may also be important.

Experimental

Preparations

2,2-Dimethyl-1,3,2-dithiastannolane (III, R = Me) was prepared by treating dipotassium ethane-1,2-dithiolate in ethanol/benzene (3/1) with dimethyltin dichloride; m.p. 78–82 °C (lit. [19] 80–81 °C), *m/e* 242. Found: C, 20.0; H, 4.1; S, 26.9. $C_4H_{10}SSn$ calcd.: C, 19.95; H, 4.2; S, 26.6%. NMR and Mössbauer data are given in Table 3.

Diethyl- and diisopropyl-dithiastannolane were prepared by heating equimolar amounts of the corresponding dichlorides, ethane-1,2-dithiol, and pyridine, in ethanol/benzene (3/1), under reflux for 4 h. III (R = Et), m.p. 100–103 °C (from pentane) (Lit. [6] 111 °C). Found: C, 26.6; H, 5.10; S, 23.5. $C_6H_{14}S_2Sn$ calcd.: C, 26.8; H, 5.24; S, 23.9%. III (R = Pr^i), m.p. 31–33 °C (from pentane).

Dibutyldithiastannolane (III, R = Bu) was prepared by heating under reflux in xylene (250 cm^3), a mixture of dibutyltin oxide (2.42 g, 0.01 mol). The reaction was probably complete within 4 h, though heating was continued for 36 h. Dimethyltin oxide (2.14 g was recovered by filtration, and the xylene was removed under vacuum. Pentane was added to the residual yellow oil, yielding the crystalline dibutyldithiastannolane, m.p. 58° (lit. [6] 59 °C). Found: C, 36.6; H, 6.6; S, 19.8. $C_{10}H_{22}S_2Sn$ calcd.: C, 36.9; H, 6.8; S, 19.8%.

Spirobis(ethane-1,2-dithiolato)tin (III) was prepared from the reaction between dipotassium ethane-1,2-dithiolate and tin(IV) tetrachloride in ethanol/benzene (3/1). M.p. 178 °C (lit. [20] 182 °C) Found: C, 15.9; H, 2.5; S, 42.5. $C_4H_8S_4Sn$ calcd.: C, 15.9; H, 2.7; S, 42.3%.

NMR spectra

Solution spectra were recorded on a Varian XL200 instrument. Solid state spectra were recorded on the ULIRS Bruker MSL300 instrument at Royal Holloway and Bedford New College, operating at frequency of 75.468 MHz for ^{13}C, 109.940 MHz for ^{117}Sn, or 111.914 MHz for ^{119}Sn. At this last frequency we experienced apparent interference from the radio contact system from the nearby Heathrow Airport, but this was absent at 109.940 MHz, hence most of solid state spectra were run on the ^{117}Sn isotope. Typical conditions were: rotation rate ca. 4500 Hz, number of transients 200–500, pulse delay ca. 10 s, contact time 1–10 ms. The suite of techniques of cross polarization, magic angle spinning, and high power decoupling were employed.

Tin chemical shifts are quoted in reference to $\delta(Me_4Sn) = 0$, and the magnetic field was calibrated with $(Me_3Sn)_4C$, $\delta + 48.2$.

Mössbauer spectra

Mössbauer spectra were recorded on the ULIRS instrument at Birkbeck College. Samples were cooled in liquid nitrogen, and isomer shifts are quoted relative to SnO_2.

X-ray crystallography

Crystal data $C_{10}H_{22}S_2Sn$, $M = 325.1$, monoclinic, a 17.978(4), b 7.886(2), c 9.974(12) Å, β 97.66(5)°, V 1401.4 Å^3, $Z = 4$, D_c 1.541 g cm^{-3}, $F(000) = 656$, space group $C2/c$.

Data collection

Unit cell parameters were measured on CAD4 diffractometer from 25 accurately centred reflexions in the angular range $10° < \theta < 14°$. Intensity data were collected from a crystal, size 0.3mm × 0.3mm × 0.2mm in the ω–2θ mode, scan speed 3.33°/min. yielding a total of 1094 reflexions of which 758 had $I > 3\sigma(I)$. A severe decay loss of intensity of 41.8% during the data collection period was corrected for and the choice of space group $C2/c$ made on the basis of the $N(z)$ test.

Structure solution and refinement

The hk(odd l) reflexions were systematically very weak which suggested that the Sn atom was lying on the centre of symmetry. Such a position would be impossible for a tetrahedral coordination around the Sn atom and inspection of the Patterson map vectors showed that the Sn atom was sited upon the diad axis with a y-coordinate close to zero. The position of the S atom was also determined.

The remainder of the structure was obtained from the first structure factor/Fourier calculation and isotropic refinement converged at $R = 0.070$ with hydrogen atoms in fixed calculated positions (d(C–H) 1.0 Å). After an absorption correction applied by the program DIFABS [21] R fell to 0.054, maximum and minimum absorption corrections 1.196 and 0.784. Full matrix anisotropic refinement of all non-hydrogen atoms (hydrogens still fixed) reached final convergence at $R = 0.032$ $R_W = 0.047$, e.s.d. of unit weight 1.071, weighting scheme $\omega^{-1} = [\sigma^2(F_0) + (0.07F)^2]^{\frac{1}{2}}$. The highest residual peak in the difference Fourier map was 0.4 electrons.

Tables of positional parameters and temperature factors are available from the Director of the Cambridge Crystallographic Data Centre, University Chemical Laboratory, Lensfield Road, Cambridge CB2 1EW (UK).

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