STRUCTURAL STUDIES IN MAIN GROUP CHEMISTRY

XXVI *. THE REACTION BETWEEN DI-μ_3-OXOBIS[μ-CHLORODIETHYLTIN(IV)]-BIS[o-CHLORODIETHYLTIN(IV)] AND 2,2'-BIPYRIDYL

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Summary

The reaction between [ClEt$_2$SnOSnEt$_2$Cl]$_2$ and 2,2'-bipyridyl has been shown to give Et$_2$SnCl$_2$ · bipyridyl and the tristannoxane adduct [ClEt$_2$SnOEt$_2$-SnOSnEt$_2$Cl] · $\frac{1}{2}$ bipyridyl. Tin-119 Mössbauer data for the tristannoxane indicate a trigonal bipyramidal geometry for all tin atoms.

Crystals of Et$_2$SnCl$_2$ · bipy are monoclinic, space group $P2_1/n$ with $a =$ 18.803(1), $b = 14.549(6)$, $c = 8.294(2)$ Å, and $\beta = 134.47(2)°$. The complex has the expected octahedral geometry with *trans* ethyl groups and mutually *cis* chlorines.

Introduction

The reactivity of dichlorotetraalkyldistannoxanes has been little investigated. Sakai [2] has reported the particularly low reactivity of the associated distannoxanes [ClEt$_2$SnOSnEt$_2$Cl]$_2$ and [ClEt$_2$SnOSnEt$_3$]$_2$ towards various cyclic lactones, in which 1 : 1 adducts of the appropriate chloroethyldistannoxane and ring-opened lactone are formed. The reaction between [ClBu$_2$SnO-SnBu$_2$Cl]$_2$ and 2,2'-bipyridyl has been briefly investigated by Davies [3] in an attempt to characterise a cyclic oligomeric dialkyltin oxide, analogous to those in organosilicon and organogermanium chemistry, by the removal of two units of dialkyltin dihalide (as its bipyridyl adduct) from one unit of dimeric distannoxane. Bu$_2$SnCl$_2$ · bipy was produced in low (15%) yield, but the reac-

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* For part XXV see ref. 1.

tion residue after removal of the solvent was found to melt in the range 60—120°C, and consequently not identified.

In an attempt to characterise the secondary product of this type of reaction, the reaction of $[ClEt_2SnOSnEt_2Cl]_2$ and 2,2'-bipyridyl has been investigated.

Experimental

$[ClEt_2SnOSnEt_2Cl]_2$ was prepared by the method of Johnson [4] and recrystallised twice from dry benzene before use. Found: C, 22.20; H, 4.74. $C_8H_{20}Cl_2OSn_2$ calcd.: C, 21.85; H, 4.59%.

The reaction of $[ClEt_2SnOSnEt_2Cl]_2$ with 2,2'-bipyridyl

To a solution of $[ClEt_2SnOSnEt_2Cl]_2$ (2.45 g, 0.006 mol) in dry benzene (10 ml) under an argon atmosphere was added a solution of 2,2'-bipyridyl (0.88 g, 0.006 mol) also in dry benzene (5 ml). The mixture was stirred at room temperature for 48 hours, during which time a small amount of white precipitate was formed. This was filtered off under an inert atmosphere, and the filtrate allowed to stand. Subsequent products crystallizing from the filtrate were similarly isolated every 24 hours. Microanalytical and melting point data for the products thus obtained are given in Table 1 and corresponding Mössbauer data in Table 2.

The crystal structure determination of $Et_2SnCl_2 \cdot bipy$
Crystal preparation

Single crystals of the title compound for intensity measurements were obtained from the reaction of 1,1,3,3-tetraethyl-1,3-dichlorodistannoxane with 2,2'-bipyridyl and used without further purification. A single crystal of approx-

TABLE 1

MICROANALYTICAL AND MELTING POINT DATA FOR PRODUCTS OF THE REACTION BETWEEN $(ClEt_2SnOSnEt_2Cl)_2$ AND 2,2'-Bipyridyl

	Found (%)			Melting point (°C)	Product
	C	H	N		
(1)	41.34	4.69	6.59	200—201	Et_2SnCl_2.bipy [a]
(2)	41.13	4.64	6.31	186—192	Et_2SnCl_2.bipy plus small amount of tristannoxane adducts as impurity.
(3)	31.80	5.31	2.94	Melting begins at 160 completed in range 188—194.	Et_2SnCl_2.bipy plus increased amount of tristannoxane adduct.
(4)	30.58	5.24	2.70	156—159, leaving a speck of solid.	Tristannoxane adduct plus small amount of Et_2SnCl_2.bipy as impurity.
(5)	28.17	5.05	1.82	162—164	Tristannoxane adduct, $ClEt_2SnOSnEt_2.OSnEt_2Cl.\frac{1}{2}$bipy [b]

[a] Independently prepared by the mixing of benzene solutions of diethyltin dichloride and 2.2'bipyridyl, recrystallised from hot benzene. $C_{14}H_{18}Cl_2N_2Sn$ calcd.: C, 41.63; H, 4.50; N, 6.93%. [b] $C_{17}H_{34}Cl_2NO_2Sn_3$ calcd.: C, 28.69; H, 4.83; N, 1.97%.

TABLE 2

MÖSSBAUER DATA [a] FOR THE PRODUCTS [b] OF THE REACTION BETWEEN $(ClEt_2SnOSnEt_2Cl)_2$ AND 2,2'-Bipyridyl

	I.S._1 [c,d]	Q.S._1 [c,d]	I.S._2 [c,d]	Q.S._2 [c,d]
(1)	—	—	1.60	3.98
(2)	—	—	1.62	3.98
(3)	1.33	3.26	1.63	3.85
(4)	1.31	3.13	1.65	3.87
(5)	1.30	3.16	—	—

[a] Measured at 77K. [b] Assignment of products 1—5 as in Table 1. [c] In mms^{-1}. [d] Subscripts 1 and 2 refer the low field and high field quadrupole split resonances, respectively. [e] P(1) refers to the area under the unresolved low field doublet. P(2) to the combined area under the high field peak and shoulder.

imate dimensions 0.3 × 0.3 × 0.2 mm, stable to both air and X-rays was mounted on a glass fibre and used for initial photography and subsequent intensity data.

Crystal data

$C_{14}H_{18}Cl_2N_2Sn$; $M = 1615.64$; $a = 18.803(1)$, $b = 14.459(6)$, $c = 8.294(2)$ Å, $\beta = 134.47(2)°$; $U = 1619.35$ Å^3, $Z = 4$, $F(000) = 800$, $\mu(Mo\text{-}K_\alpha) = 19.02$ cm^{-1}. Space group $P2_1/n$ by systematic absences ($h0l$ for $h + l = 2n + 1$ and $0k0$ for $k = 2n + 1$).

The space group and initial cell parameters were determined from oscillation and zero- and first-layer Weissenberg photographs. Relative intensities up to $\theta = 25.0°$ were collected with Mo-K_α radiation using a Hilger and Watts Y290 four-circle automatic diffractometer. Accurate cell dimensions were obtained by

TABLE 3

FINAL FRACTIONAL ATOMIC COORDINATES IN Et_2SnCl_2·2,2'-bipy (ESTIMATED STANDARD DEVIATIONS IN PARENTHESES)

Atom	x/a	y/b	z/c
Sn(1)	0.21176(2)	0.21489(2)	0.15768(5)
Cl(1)	0.3533(1)	0.3295(1)	0.3946(2)
Cl(2)	0.2502(1)	0.1016(1)	0.4441(2)
N(1)	0.1324(3)	0.2917(3)	−0.1848(7)
N(2)	0.0674(3)	0.1333(3)	−0.1581(7)
C(1)	0.1252(4)	0.2914(4)	0.1880(10)
C(2)	0.0981(4)	0.3895(5)	0.0980(10)
C(3)	0.2972(4)	0.1290(4)	0.135(1)
C(4)	0.3122(4)	0.1650(4)	−0.0070(10)
C(5)	0.1665(4)	0.3729(4)	−0.1860(9)
C(6)	0.1146(3)	0.4234(3)	−0.3792(8)
C(7)	0.0265(3)	0.3910(4)	−0.5776(8)
C(8)	−0.0099(5)	0.3070(5)	−0.5800(10)
C(9)	0.0458(6)	0.2599(5)	−0.3770(10)
C(10)	0.0109(8)	0.1693(5)	−0.3650(2)
C(11)	−0.0736(5)	0.1254(4)	−0.5550(10)
C(12)	−0.1002(8)	0.0422(6)	−0.5280(20)
C(13)	−0.0415(4)	0.0043(4)	−0.3160(10)
C(14)	0.0422(5)	0.0521(4)	−0.1340(10)

TABLE 4

FINAL ANISOTROPIC THERMAL PARAMETERS IN $Et_2SnCl_2.2,2'$-bipy (ESTIMATED STANDARD DEVIATIONS IN PARENTHESES)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Sn(1)	3.85(2)	4.49(2)	3.37(2)	0.02(1)	2.05(2)	—0.37(1)
Cl(1)	5.7(5)	6.5(4)	4.6(9)	—0.6(8)	2.3(4)	—2.3(2)
Cl(2)	6.4(3)	7.9(1)	4.6(2)	1.4(6)	2.6(5)	—1.6(7)
N(1)	4.4(2)	4.7(2)	3.8(2)	0.2(2)	2.4(2)	—0.1(2)
N(2)	4.2(2)	4.6(2)	4.1(2)	—0.6(2)	2.6(2)	—0.8(2)
C(1)	8.9(3)	7.1(3)	9.4(3)	—0.8(3)	7.4(3)	0.3(2)
C(2)	13.5(3)	6.3(4)	16.3(4)	1.0(3)	12.0(3)	2.3(3)
C(3)	5.8(3)	6.0(3)	8.0(4)	0.6(3)	4.8(3)	0.9(3)
C(4)	12.8(3)	9.6(3)	14.2(4)	—0.1(3)	11.5(3)	1.4(2)
C(5)	6.4(3)	5.1(3)	5.7(3)	0.7(2)	4.1(3)	0.0(3)
C(6)	6.7(3)	6.3(3)	5.9(2)	1.5(2)	4.2(2)	0.5(2)
C(7)	7.6(2)	5.6(3)	5.5(2)	1.7(2)	4.1(2)	1.2(2)
C(8)	5.3(4)	6.5(3)	3.6(3)	0.6(3)	2.2(3)	0.9(3)
C(9)	4.2(5)	4.7(4)	3.6(5)	0.1(3)	2.4(5)	0.6(3)
C(10)	3.7(7)	4.7(4)	4.0(9)	—0.3(5)	2.3(8)	0.2(5)
C(11)	4.6(3)	6.3(3)	4.9(4)	—1.1(3)	2.6(3)	—0.3(3)
C(12)	4.9(7)	7.0(6)	7.3(8)	—2.1(6)	3.9(7)	—1.4(5)
C(13)	5.5(3)	6.4(3)	6.5(3)	—1.3(2)	4.2(3)	—1.3(3)
C(14)	5.6(4)	5.3(4)	6.7(3)	—0.4(3)	4.3(3)	—1.0(3)

U_{ij} are of the form $10^2 \exp - 2\pi^2(h^2 U_{11}a^{*2} + k^2 U_{22}b^{*2} + 1U_{33}c^{*2} + 2hkU_{12}a^*b^* + 2hlU_{13}a^*c^* + 2klU_{23}b^*c^*)$

least-squares refinement of ca. 20 reflections. The reflections were brought to the same relative intensities by reference reflections taken every 100 reflections. Background counts were low and included in the usual manner. All reflections with $I < 3\sigma(I)$ were considered non-observed, reducing the number of reflection from 3000 to 2584. The intensities were corrected for Lorentz and polarisation effects, no absorption correction being made due to the low μ value.

Structure determination and refinement

The positional parameters of the tin atom in the asymmetric unit were determined by Patterson methods, and were used to phase the initial structure factor calculation. After one cycle of full matric isotropic least-squares refinement of these positional parameters, a Fourier synthesis yielded the positions of all the outstanding (light) atoms.

Four cycles of full matrix, isotropic least-squares refinement and four cycles of full matrix anisotropic least-squares refinement of the positional parameters of all atoms, yielded a convergence of the R value at 0.0493.

At this point, a weighting scheme based on a Chebychev series [5] in five terms, coefficients 287.2, 454.5, 248.2, 98.8, and 28.3, was introduced. A subsequent three cycles of full matric, anisotropic least-squares refinement gave a final R value of 0.0407.

All calculations were performed using the CRYSTALS suite of programmes [6]. Scattering factors used were those for neutral atoms [7] *.

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* Tables of observed and calculated structure factors are available from the author on request.

TABLE 5 251

FINAL INTRAMOLECULAR BOND DISTANCES (Å) AND ANGLES (deg) IN Et$_2$SnCl$_2$.2,2'-bipy
(ESTIMATED STANDARD DEVIATIONS IN PARENTHESES)

(a) *Distances*

Sn(1)—Cl(1)	2.529(1)	N(1)—C(5)	1.348(8)
Sn(1)—Cl(2)	2.545(1)	N(1)—C(9)	1.330(8)
Sn(1)—N(1)	2.368(4)	C(5)—C(6)	1.367(7)
Sn(1)—N(2)	2.382(4)	C(6)—C(7)	1.362(7)
Sn(1)—C(1)	2.127(6)	C(7)—C(8)	1.392(8)
Sn(1)—C(3)	2.146(6)	C(8)—C(9)	1.391(10)
C(1)—C(2)	1.523(8)	C(9)—C(10)	1.507(10)
C(3)—C(4)	1.484(8)		
		N(2)—C(14)	1.339(7)
		N(2)—C(10)	1.340(12)
		C(10)—C(11)	1.381(12)
		C(11)—C(12)	1.389(11)
		C(12)—C(13)	1.374(12)
		C(13)—C(14)	1.383(9)

(b) *Angles*

Cl(1)—Sn(1)—Cl(2)	104.2(3)	N(1)—Sn(1)—N(2)	69.0(1)
Cl(1)—Sn(1)—N(1)	93.2(1)	N(1)—Sn(1)—C(1)	90.2(2)
Cl(1)—Sn(1)—N(2)	162.0(1)	N(1)—Sn(1)—C(2)	92.8(2)
Cl(1)—Sn(1)—C(1)	92.2(2)	N(2)—Sn(1)—C(1)	90.8(2)
Cl(1)—Sn(1)—C(3)	90.4(2)	N(2)—Sn(1)—C(2)	87.7(2)
Cl(2)—Sn(1)—N(1)	162.5(1)	C(1)—Sn(1)—C(2)	175.8(2)
Cl(2)—Sn(1)—N(2)	93.6(1)		
Cl(2)—Sn(1)—C(1)	88.2(2)	Sn(1)—C(1)—C(2)	116.9(4)
Cl(2)—Sn(1)—C(2)	88.0(2)	Sn(1)—C(3)—C(4)	115.8(4)
Sn(1)—N(1)—C(5)	121.3(4)	Sn(1)—N(2)—C(10)	118.9(4)
Sn(1)—N(1)—C(9)	119.2(4)	Sn(1)—N(2)—C(14)	121.7(4)
C(5)—N(1)—C(9)	119.0(5)	C(14)—N(2)—C(10)	119.4(5)
N(1)—C(5)—C(6)	121.8(5)	N(2)—C(10)—C(11)	121.8(6)
C(5)—C(6)—C(7)	119.7(5)	C(10)—C(11)—C(12)	118.4(8)
C(6)—C(7)—C(8)	119.5(5)	C(11)—C(12)—C(13)	119.9(8)
C(7)—C(8)—C(9)	117.9(6)	C(12)—C(13)—C(14)	118.4(6)
C(8)—C(9)—N(1)	122.2(6)	C(13)—C(14)—N(2)	122.1(5)
C(8)—C(9)—C(10)	121.2(8)	N(2)—C(10)—C(9)	115.8(8)
N(1)—C(9)—C(10)	116.6(7)	C(11)—C(10)—C(9)	122.3(8)

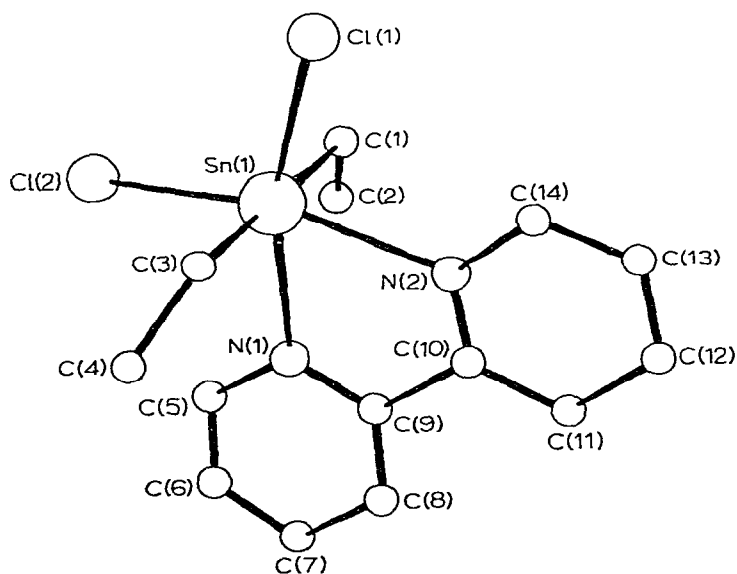Fig. 1. The structure of diethyltin dichloride 2,2'-bipyridyl complex, showing atomic numbering.
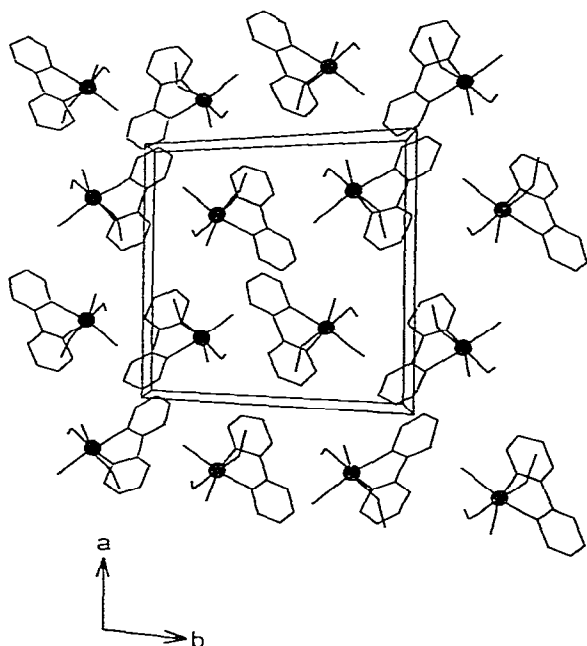
Fig. 2. Projection of the unit cell of $Et_2SnCl_2 \cdot 2,2'$-bipyridyl onto the ab plane.

Final fractional atomic coordinates are given in Table 3, final anisotropic thermal parameters in Table 4, and intramolecular bond distances and angles in Table 5. The molecular geometry, with atomic labelling is shown in Fig. 1, and the projection of the unit cell on to the ab plane in Fig. 2.

Discussion

Mössbauer, analytical and melting point data for the reaction of $[ClEt_2SnO-SnEt_2Cl]_2$ and 2,2'-bipyridyl, collected in Tables 1 and 2 and shows in Fig. 3, show that the initial product of the reaction is $Et_2SnCl_2 \cdot$ bipy. Subsequent crystallisation fractions comprise a mixture of $Et_2SnCl_2 \cdot$ bipy together with a second product (Fig. 3, B and C), before a final fraction consisting of the pure second component, identified by analysis as the tristannoxane adduct, $ClEt_2Sn-OSnEt_2Cl \cdot \frac{1}{2}$ bipy, is obtained (Fig. 3, D). Although apparently pure $Et_2SnCl_2 \cdot$ 2,2'-bipy from its Mössbauer spectrum, fraction 2 must contain a trace of the second reaction product, since its melting point (186—192°C) is substantially lower than that of the pure compound prepared independently (200—201°C).

The Mössbauer spectrum of the pure second product, $ClEt_2SnOSnEt_2Cl \cdot \frac{1}{2} \cdot$ 2,2'-bipy, consists of only one quadrupolar-split doublet (I.S. = 1.30 mm s^{-1}, Q.S. = 3.16 mm s^{-1}), indicating that all the tin environments are very similar These data resemble very closely those for $[ClMe_2SnOSnMe_2Cl]_2$ (I.S. = 1.28 mm s^{-1}, Q.S. = 3.17 mm s^{-1}) and (less so) $[ClEt_2SnOSnEt_2Cl]_2$ (I.S. = 1.50 mm s^{-1}, Q.S. = 3.4 mm s^{-1}) [8]. In the former compound, both tin atoms have a trigonal bipyramidal environment with the two methyl groups occupying equatorial positions. The structure of $[ClEt_2SnOSnEt_2Cl]_2$ was only partially deter-
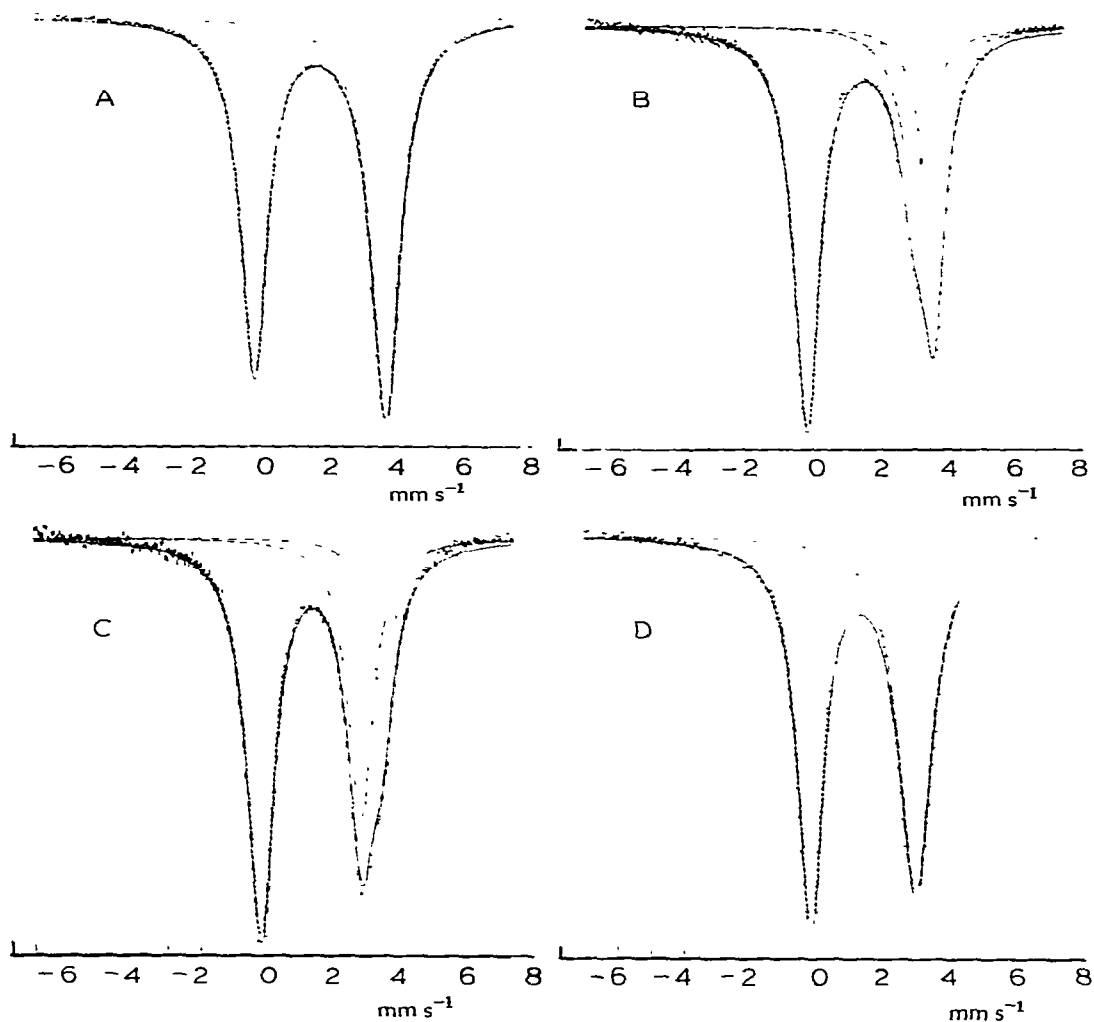
Fig. 3. Tin-119 Mössbauer spectra for crystallisation fractions 2 (pure $Et_2SnCl_2 \cdot 2,2'$-bipy) (A), 3 (B), 4 (C), and 5 (pure $ClEt_2SnOSnEt_2OSnEt_2Cl \cdot \frac{1}{2}$ bipy) (D).

mined due to disorder, but was similar to that of the methyl analogue [8]. It would appear that the tin environments in the tristannoxane-bipyridyl adduct have very similar five-coordinated, trigonal bipyramidal tin environments.

The formation of both diethyltin dichloride bipyridyl and the tristannoxane moiety in the title reaction can be readily interpreted in the light of the structure of the distannoxane, determined by X-ray crystallography [8]. Although some dissociation of the dimeric distannoxane might be expected to occur in solution, some dimeric species will remain, as demonstrated by the results of Pfeiffer and Brack [9], who determined the value of n in the formula $[ClEt_2Sn-OSnEt_2Cl]_n$ in freezing benzene to be in the range 1.4–1.8 and independent of concentration.

Assuming the reactive dimeric species to be the major contributor to the disordered solid state structure of the symmetrical dichlorotetraethyldistannoxane (Fig. 4 (A)), and that this structure is essentially maintained in solution, the
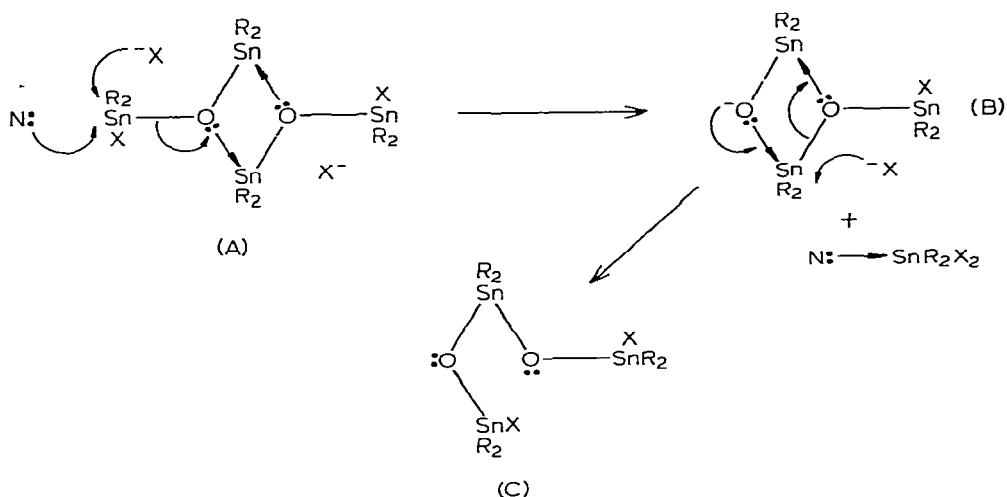
Fig. 4. Proposed mechanism for the reaction of [ClEt$_2$SnOSnEt$_2$Cl]$_2$ and 2,2-bipyridyl.

formation of diethyltin dichloride bipyridyl is readily explained in the light of the presence of a (presumably) labile chlorine anion. Indeed, the lability of the halide is endorsed by the presence of only one broad ^{19}F resonance in the magnetic resonance spectrum of (Bu$_4$Sn$_2$F$_2$O)$_2$ in benzene. This can be interpreted as very rapid fluorine exchange, viz:
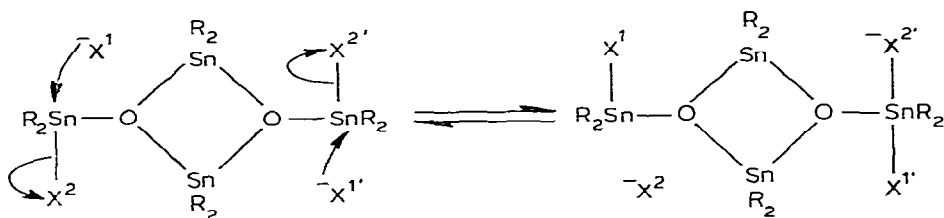
Rearrangement of the resulting cyclic tristannoxane, (B, Fig. 4), will yield the linear tristannoxane (C, Fig. 4). No unequivocal structural evidence is available for the linearity of the tristannoxane or for the mode of coordination of the hemi-bipyridyl unit. No suitable single crystals of the tristannoxane adduct could be obtained, precluding X-ray analysis. The only literature report of a structure related to the proposed product is the partially determined structure of hexa-n-butyl-1,5-diazidotristannoxane, for which the centrosymmetric dimeric ladder structure (Fig. 5) was inferred [10]. Molecular weight measurements suggest that in dilute solution the tristannoxane, ClSnBu$_2$OSnBu$_2$Cl, is essentially monomeric, although at high concentration, some association may occur [11].

Crystal and molecular structure of Et$_2$SnCl$_2$ · bipy

Diethyltin dichloride bipyridyl has a molecular octahedral six-coordinate *trans*-ethyl, *cis*-N.N, *cis*-Cl geometry, and a lattice consisting of monomeric non-interacting Et$_2$SnCl$_2$ · bipy units.
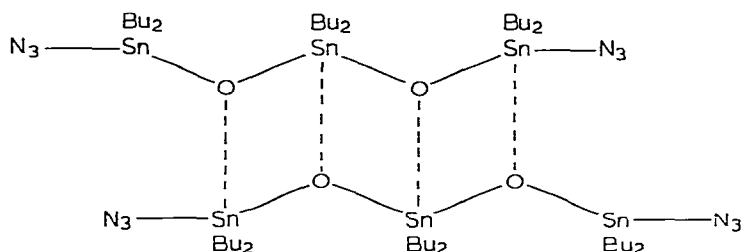
Fig. 5. Proposed structure for [N₃Bu₂SnOSnBu₂OSnBu₂N₃]₂ [12].

A comparison of Sn—C(Et), Sn—Cl and Sn—M bond distances in the title compound with corresponding distances in related compounds, is given in Table 6. Since no six-coordinated diethyltin species have yet been characterised by X-ray crystallography, no direct comparison of Sn—C(Et) bond lengths is possible. The Sn—C(Et) bond lengths are, however, compatible with the general trend of reported Sn—C(Me) bond lengths (2.08(5)—2.22(2) Å) and are shorter than the corresponding Sn—C(Ph) distance in Ph_2SnCl_2 · bipy (2.152(8) Å). The Sn—Cl distance in the title compound (2.529(1), 2.545(1) Å) is marginally longer than the corresponding distance in Ph_2SnCl_2 · bipy (2.509(2) Å), and, in conjunction with the shortening of the Sn—C bond in the ethyl derivative, reflects a preferential increase in the 5s content of the Sn—C(Et) bonds over the Sn—Cl bonds. This is reflected not only in the increase in the C—Sn—C angle from 173.5(3)° in Ph_2SnCl_2 · bipy to 175.8(2)° in Et_2SnCl_2 · bipy, but also in an increase in the I.S. parameters (1.35 mms⁻¹ for Ph_2SnCl_2 · bipy) to 1.60 mms⁻¹ for Et_2SnCl_2 · bipy). Since only small changes in the C—Sn—C angle occur in the comparison of the two bipyridyl adducts, the large change in the I.S. values will be explained by a second, complementary factor, the —I effect of the ethyl group compared to the +I effect of the phenyl group. The Sn—N distance (2.368(4), 2.382(4) Å) is very similar to the corresponding distances in Ph_2SnCl_2 · bipy (2.344(6), 2.375(6) Å).

Distortions of a regular octahedral geometry are observed. Thus, while the C—Sn—C angle (175.8(2)°) is close to the regular angle (180°), the bite of the

TABLE 6

COMPARISON OF INTRAMOLECULAR BOND DISTANCES (Å) IN Et_2SnCl_2.bipy WITH THOSE IN RELATED COMPOUNDS [a]

Compound	Sn—C	Sn—Cl	Sn—N	Reference
Et_2SnCl_2.bipy	2.127(6)	2.529(1)	2.368(4)	This work
	2.146(6)	2.545(1)	2.382(4)	
Ph_2SnCl_2.bipy	2.152(8)	2.509(2)	2.344(6)	12
			2.375(6)	
Me_2SnCl_2	2.210(80)	2.400(40)		13
$Me_2Sn(acac)_2$	2.140(20)			14
Me_2SnCl_2.2pyO	2.220(20)	2.580(10)		15
Me_2SnCl_2.2 DMSO	2.070(60)	2.480(10)		16
	2.080(50)	2.530(10)		

[a] All have octahedral, trans-[R₂SnX₄] geometry.

bidentate bipyridyl ligand reduces the N—Sn—N angle to 69.0(1)°, and mutual repulsion of the chlorine atoms causes an opening of the Cl—Sn—Cl angle to 104.2(3)°. These angles compare favourably with the corresponding angles (173.4(3)°, 69.0(2)° and 103.5(1)° respectively) in $Ph_2SnCl_2 \cdot$ bipy.

The angles at the α-carbon (116.9(4), 115.8(4)°) are somewhat larger than expected for a tetrahedrally coordinated carbon atom, presumably due to the increased s-character of the Sn—C(Et) bonds. The high primary anisotropic thermal parameters U_{11}, U_{22} and U_{33} of the β-carbons C(2) (13.5(3), 6.3(4),) and C(4) (12.8(3), 9.6(3) and 14.2(4) reflects a degree of disorder associated with the terminal carbon of an alkyl chain. The C—C bond distances within the ethyl groups (1.523(8), 1.484(8) Å) are somewhat smaller than expected (1.541(3), (183)), probably as a consequence of this disorder. The disposition of the β-carbons is similar, both being bent towards the plane of the bipyridyl residue. This is presumably a consequence of electronic repulsion by the electronegative axial chlorine atom (Cl(1)).

The maximum deviation of atoms (N(1), N(2), C(5)—C(14)) from the mean plane through these atoms is 0.12 Å, reflecting the essential planarity of bipyridyl residue. This plane can also be considered as the plane containing all the equatorial atoms (maximum deviation from this plane, Cl(2), 0.35 Å).

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