Mössbauer spectroscopic investigations of intra- and intermolecularly coordinated organotin compounds

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Abstract

The Mössbauer parameters are reported on twenty-five tetra-, penta- and hexacoordinated organotin compounds of the general types $\mathrm{RSn(CH_2CH_2CH_2)_3N}$ (R = Cl, Me), $\mathrm{RR'Sn(CH_2CH_2CH_2)_2E}$ (R = Cl, Me; R' = Cl, Me; E = NMe, O), $\mathrm{Me_2ClSnCH_2CH_2CH_2E}$ ($\mathrm{E = NMe_2, PPhBu^t, MeNCH_2CH_2N(Me)CH_2CH_2CH_2SnMe_2Cl}$), $\mathrm{RR'Sn[CH_2CH_2CH_2N(Me)CH_2]_2}$ (R = R' = Cl, Br, I, SPh; R = Me, R' = Cl), $\mathrm{\{SSn[CH_2CH_2CH_2N(Me)CH_2]_2\}_2}$, $\mathrm{Cl_2Sn(CH_2CH_2CH_2NMe_2)_2}$, $\mathrm{S(CH_2CH_2CH_2SnMe_{(3-n)}Cl_n)_2}$ ($n = 1,2$), $\mathrm{Et_{(3-n)}X_nSnCH_2CH_2E}$ ($n = 0$, E = P(O)(OEt)Ph, $\mathrm{P(O)Bu_2}$; $n = 1$, X = Br, E = P(O)(OEt)Ph, $\mathrm{P(O)Bu_2}$), $\mathrm{Me_2ClSnCH_2CH_2PPh_2}$ and $\mathrm{Ph(O)P(CH_2CH_2SnClMe_2)_2}$. Quadrupole splitting and η values calculated on the basis of the concept of partial quadrupole splittings are in agreement with the molecular structures concluded from X-ray measurements and NMR studies.

Introduction

Since the discovery of the Mössbauer effect, Mössbauer spectroscopy has been widely used as a powerful tool for the elucidation of structural problems relating to organotin compounds [1, 2]. With this technique, information is readily obtained about the oxidation state and the coordination sphere of the metal atom. Through use of the concept of partial quadrupole splitting (PQS), different isomers of penta- and hexacoordinated structures have been distinguished [3–6].

In recent years, we have prepared a number of organotin compounds of the types shown in Scheme 1, which were characterized by NMR and X-ray investigations. These studies unambiguously confirmed the existence of intra- or intermolecular donor–acceptor interactions in most of the derivatives. In this paper, we present Mössbauer parameters, together with model calculations on these compounds, based on the PQS concept, in order to compare them with structural findings concluded from X-ray and NMR studies.

Results and discussion

Table 1 summarizes the experimental isomer shifts, *IS*, the experimental and calculated quadrupole splittings, QS_{exp} and QS_{calc}, and the calculated asymmetry parameters η.

PQS model calculations of QS_{calc} and η were performed according to published procedures [1, 3]. For trigonal bipyramidal structures, the following PQS values (mm/s) were used [3, 4] the superscripts tba and tbe refer to trigonal bipyramidal apical and equatorial, respectively: $\{\mathrm{Cl}\}^{\mathrm{tba}}$ 0.00, $\{\mathrm{Cl}\}^{\mathrm{tbe}}$ +0.20; $\{\mathrm{alkyl}\}^{\mathrm{tba}}$ −0.94; $\{\mathrm{alkyl}\}^{\mathrm{tbe}}$, $\{\mathrm{CH_2}\}^{\mathrm{tbe}}$ −1.13; $\{\mathrm{N}\}^{\mathrm{tba}}$ +0.01; $\{\mathrm{N}\}^{\mathrm{tbe}}$ +0.21 (this corresponds to the value for apical piperidine); $\{\mathrm{O}\}^{\mathrm{tba}}$ −0.21; $\{\mathrm{PPhBu^t}\}^{\mathrm{tba}}$ +0.095 (this value was calculated from compound **9**); $\frac{1}{2}\{\mathrm{S}\}^{\mathrm{tba}}$ +0.135 (half of the value calculated for the apical donating sulfur in $\mathrm{Cl_2Sn(SCH_2CH_2)_2S}$ [4]); $\{\mathrm{RR'PO}\}^{\mathrm{tba}}$ +0.12 (the value for $\{\mathrm{Ph_3PO}\}^{\mathrm{tba}}$).

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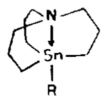
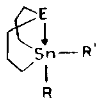
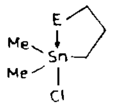


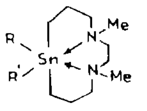
1, R = Cl
2, R = Me



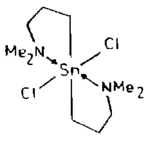
3, R = R' = Cl, E = NMe
4, R = R' = Cl, E = 0
5, R = Cl, R' = Me, E = NMe
6, R = R' = Me, E = NMe
7, R = R' = Me, E = 0



8, E = NMe_2
9, E = $P(Ph)Bu^t$
10, E = $MeNCH_2CH_2N(Me)CH_2CH_2CH_2SnMe_2C$



11, R = R' = Cl
12, R = R' = Br
13, R = R' = I
14, R = R' = SPh
15*, R = R' = S
16, R = Me, R' = Cl



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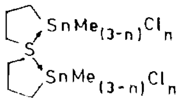

18, $n = 1$
19, $n = 2$

$R_{(3-n)}X_nSnCH_2CH_2E$

20, $n = 0$, R = Et, E = P(O)(OEt)Ph
21, $n = 0$, R = Et, E = $P(O)Bu_2$
22, $n = 1$, R = Et, E = P(O)(OEt)Ph, X = Br
23, $n = 1$, R = Et, E = $P(O)Bu_2$, X = Br
24, $n = 1$, R = Me, E = PPh_2, X = Cl

$Ph(O)P(CH_2CH_2SnMe_2Cl)_2$
25

*$\{SSn[CH_2CH_2CH_2N(Me)CH_2]_2\}_2$

Scheme 1.

The PQS model equations for the principal components of the EFG tensor in regular trigonal bipyramidal structures (Fig. 1) are as follows:

(A)

$$V_{zz} = 2\{N\}^{tba} + 2\{R\}^{tba} - 3\{CH_2\}^{tbe}$$

$$\eta = 0$$

(B)

$$V_{kk} = 2\{E\}^{tba} + 2\{R\}^{tba} - \{R'\}^{tbe} - 2\{CH_2\}^{tbe}$$

$$V_{jj} = -\{E\}^{tba} - \{R\}^{tba} + 2\{R'\}^{tbe} - 0.5\{CH_2\}^{tbe}$$

$$V_{ii} = -\{E\}^{tba} - \{R\}^{tba} - \{R'\}^{tbe} + 2.5\{CH_2\}^{tbe}$$

$$\eta = (3\{R'\}^{tbe} - 3\{CH_2\}^{tbe})/V_{zz}$$

(For **3** and **4**, V_{ii} was chosen as V_{zz}, while for **5** to **7**, $V_{kk} = V_{zz}$).

Thus for **3** and **4**

$$\eta = (3\{E\}^{tba} + 3\{R\}^{tba} - 3\{R'\}^{tbe} - 1.5\{CH_2\}^{tbe})/V_{ii}$$

and for **5**–**7**

$$\eta = (3\{R'\}^{tbe} - 3\{CH_2\}^{tbe})/V_{kk}.$$

(C), (F)

$$V_{zz} = 2\{E\}^{tba} + 2\{Cl\}^{tba} - 2\{Me\}^{tbe} - \{CH_2\}^{tbe}$$

$$V_{jj} = V_{xx} = -\{E\}^{tba} - \{Cl\}^{tba} + 2\{CH_2\}^{tbe} - 0.5\{Me\}^{tbe}$$

$$V_{ii} = V_{yy} = -\{E\}^{tba} - \{Cl\}^{tba} - \{CH_2\}^{tbe} + 2.5\{Me\}^{tbe}$$

$$\eta = (3\{CH_2\}^{tbe} - 3\{Me\}^{tbe})/V_{zz}$$

(G)

$$V_{zz} = 2\{RR'PO\}^{tba} + 2\{Br\}^{tba} - 2\{Et\}^{tbe} - \{CH_2\}^{tbe}$$

$$V_{jj} = V_{xx} = -\{RR'PO\}^{tba} - \{Br\}^{tba} + 2\{CH_2\}^{tbe} - 0.5\{Et\}^{tbe}$$

$$V_{ii} = V_{yy} = -\{RR'PO\}^{tba} - \{Br\}^{tba} - \{CH_2\}^{tbe} + 2.5\{Et\}^{tbe}$$

$$\eta = (3\{CH_2\}^{tbe} - 3\{Et\}^{tbe})/V_{zz}$$

Subsequently, the values $QS_{calc} = \frac{1}{2}eQV_{zz}\ (1 + \frac{1}{3}\eta^2)^{1/2}$ were obtained [1].

For the hexacoordinated species of types (D) and (E) (Fig. 1), the following PQS values and equations were used [1]: [alkyl], [CH_2] −1.03; [Cl], [Br] 0.00;

TABLE 1. Isomer shifts, *IS*, observed quadrupole splittings, QS_{exp}, calculated quadrupole splittings, QS_{calc} (according to the idealized structures shown in Fig. 1) and η values for **1–24**.

Compound		*IS* (mm/s)	QS_{exp} (mm/s)	QS_{calc} (mm/s)	η_{calc}
(A)	**1**	1.47	2.74	−3.41; −3.13[a]	0
	2	1.40	0.60	−1.53; −0.85[a]	0
(B)	**3**	1.56	2.98	−3.05	0.46
	4	1.64	3.31	−2.77	0.31
	5	1.47	2.99	−3.41	0
	6	1.26	0	−1.53	0
	7	1.33	0	−1.09	0
(C)	**8**	1.00	3.12	−3.41	0
	9	1.36	3.20	−3.20	0
	10	1.33	3.36	−3.41	0
(D) and (D′)	**11**	1.62	3.92	−4.10	0
	12	1.65	3.24	−4.10	0
	13	1.49	3.39	−3.84	0
	14	1.18	3.74	−3.40	0
	15	1.74	2.74	−3.43	0
	16	0.95	3.33	−3.41[b]; +3.58[c]	0[b]; 0.99[c]
(E) and (E′)	**17**	1.55	3.73	−4.14	0.014
(F)	**18**	0.95	3.13	−3.66; −3.37[a]	0
	19	0.81	3.40	−3.37	0.58
(G)	**20**	1.37	0		
	21	1.34	0		
	22	1.51	3.36	−3.63	0
	23	1.48	3.39	−3.63	0
	24	1.38	3.25	−3.39[d]; −3.20[e]	0

[a] Calculated by using the true molecular structure. [b] Calculated for structure (L), see Fig. 3. [c] Calculated for structure (M), see Fig. 3. [d] Calculated for structure (I), see Fig. 2. [e] Calculated for structure (J), see Fig. 2. Structures (D) and (D′), (E) and (E′), respectively, give practically identical *QS* values in the present system because of the small (or zero) η values for both structures. (The V_{zz} values are identical.)

[I] −0.14; [N] +0.01; [SpH] −0.37 calculated from $[\mathrm{SPh}]^{\mathrm{tetr}}$ [7] by using the relation $[\mathrm{L}]^{\mathrm{tetr}}:[\mathrm{L}]^{\mathrm{oct}} = 1:0.67$); [S] −0.355 (calculated from $[\mathrm{S}]^{\mathrm{tetr}}$ [7]).

(D)

$$V_{kk} = V_{zz} = 4\{\mathrm{CH_2}\} - 2\{\mathrm{R}\} - 2\{\mathrm{N}\}$$

$$V_{ii} = V_{jj} = V_{xx} = V_{yy} = -2\{\mathrm{CH_2}\} + \{\mathrm{R}\} + \{\mathrm{N}\}$$

$$\eta = 0$$

(D′)

$$V_{kk} = V_{zz} = 4\{\mathrm{CH_2}\} - \{\mathrm{R}\} - \{\mathrm{R'}\} - 2\{\mathrm{N}\}$$

$$V_{jj} = V_{yy} = 4\{\mathrm{N}\} - 2\{\mathrm{CH_2}\} - \{\mathrm{R}\} - \{\mathrm{R'}\}$$

$$V_{ii} = V_{xx} = 2\{\mathrm{R}\} - 2\{\mathrm{R'}\} - 2\{\mathrm{CH_2}\} - 2\{\mathrm{N}\}$$

$$\eta = (6\{\mathrm{N}\} - 3\{\mathrm{R}\} - 3\{\mathrm{R'}\})/V_{zz}$$

(E)

$$V_{kk} = V_{zz} = 4\{\mathrm{CH_2}\} - 2\{\mathrm{Cl}\} - 2\{\mathrm{N}\}$$

$$V_{ii} = V_{yy} = 4\{\mathrm{N}\} - 2\{\mathrm{CH_2}\} - 2\{\mathrm{Cl}\}$$

$$V_{jj} = V_{xx} = 4\{\mathrm{Cl}\} - 2\{\mathrm{CH_2}\} - 2\{\mathrm{N}\}$$

$$\eta = -6\{\mathrm{N}\}/V_{zz}$$

(E′)

$$V_{kk} = V_{zz} = 4\{\mathrm{CH_2}\} - 2\{\mathrm{Cl}\} - 2\{\mathrm{N}\}$$

$$V_{ii} = V_{jj} = V_{xx} = V_{yy} = -2\{\mathrm{CH_2}\} + \{\mathrm{Cl}\} + \{\mathrm{N}\}$$

$$\eta = 0$$

From a structural point of view, compounds **1–25** can be divided into three classes: tetracoordinated, pentacoordinated and hexacoordinated derivatives (Scheme 1).

Among the pentacoordinated derivatives, compounds **1**, **2**, **4**, **5** and **18** show big differences between QS_{calc} and QS_{exp}, suggesting substantial distortions from ideal geometry. Indeed, the molecular structures of **1**, **2** and **18** exhibit more or less marked deviations from the ideal trigonal bipyramidal skeleton [8–10]. When these actually observed structures were used for the calculation of *QS* values (denoted by a in Table 1), a much better fitting between QS_{calc} and QS_{exp} was observed. Within the stannaocane family **3–7**, the dichloro derivative **3** displays an acceptable

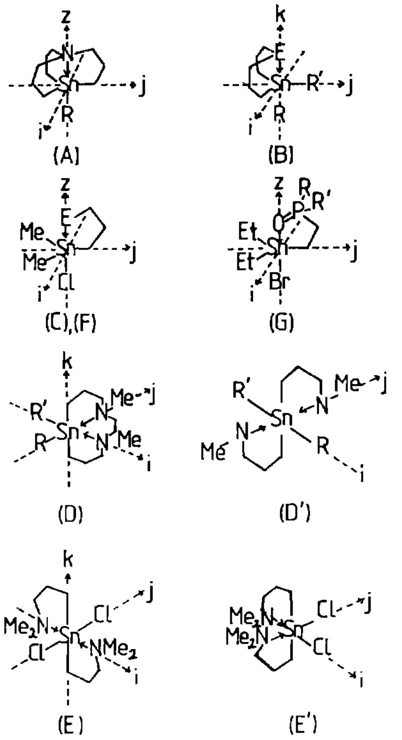

Fig. 1. Principal EFG axes for structures (A) to (G) in idealized regular trigonal bipyramidal and octahedral configurations.

agreement between QS_{exp} and QS_{calc}, which could be expected due to its solid-state structure [11].

For 6 from low-temperature ^1H and ^{13}C NMR measurements, we concluded a tin–nitrogen interaction in solution [11]. However, the Mössbauer spectrum does not show any *QS*, suggesting no or only a very small deviation from tetrahedral geometry. The same holds for compound 7.

The monocyclic derivatives 8 and 9 exhibit remarkable similar *QS* values, although trivalent phosphorus is known to be a poor donor towards tin(IV) as compared with trivalent nitrogen [12]. Obviously, this difference in donor capacity is much more expressed in their different *IS* values. For the stannaocanes of the type $Ph(Cl)Sn(SCH_2CH_2)_2E$ ($E=NMe$, $IS=1.27$, $QS=1.84$; $E=PPh$, $IS=1.63$, $QS=1.81$ mm/s), similar results were observed.

The phosphorus-containing compounds **20–24** show the expected tetrahedral (**20**, **21**) and trigonal bipyramidal (**22**, **23**) geometries. The situation is more complicated for compound **24**. QS_{exp} unambiguously suggests pentacoordination; this can not be achieved by intramolecular cyclization (formation of a four-membered cycle), but by intermolecular association via chloro or phosphorus bridges (Fig. 2). From the QS_{calc} values, these structures cannot be distinguished unambiguously. In our experience,

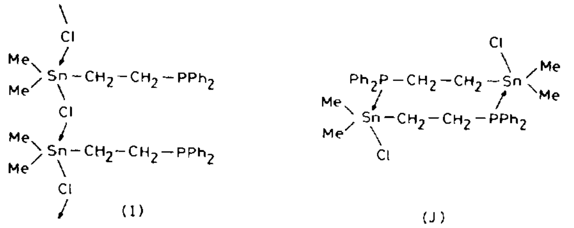

Fig. 2. Structural alternatives (I) and (J) for $Me_2ClSnCH_2CH_2PPh_2$.

the formation of an eight-membered cycle (J) is less favoured than the arrangement (I) with an infinite zig-zag Sn–Cl...Sn chain. The molecular structure of this particular compound is of interest. Compounds **11–17** are of special interest because they show big differences in their *IS* and *QS* values, which were not expected as a result of the variation of the substituents R alone. Thus, for **11** to **14** octahedral, *trans* C-configurated structures are favoured for stereochemical reasons. The dichloro, diiodo and dithiolato derivatives **11**, **13** and **14** fit in with this prediction, though the differences between QS_{calc} and QS_{exp} are relatively high for **13** and **14**. However, the dibromo derivative **12** exhibits a very small value as compared with those found for **11**, **13** and **14**. The reason could be a strong distortion of the molecular skeleton in **12**, for which we have no explanation. In the *trans* configurated bipyridyl complexes $Bu_2SnX_2 \cdot bipy$ ($X=Cl$, Br), the *QS* values amount to 3.95 mm/s and are independent of the nature of the halogen atoms [13].

The spiro compound **15** exhibits an unexpectedly small QS_{exp} with respect to QS_{calc}. The molecular structure of this particular compound shows considerable distortions from octahedral geometry. Thus, the C–Sn–C angle amounts to 156° and the Sn–N interactions are only weak and different (2.766 and 2.859 Å) [14]. From a stereochemical point of view, this molecule must be regarded as an intermediate between the transition trigonal bipyramid ↔ octahedron.

Among the compounds of type (D), the non-symmetric derivative **16** exhibits particular behaviour. From NMR measurements and also from the high solubility in water, we concluded an ionic pentavalent structure (K) (Fig. 3), but this could not be proved unambiguously [15]. Alternative structures are the trigonal bipyramidal arrangement (L) and the octahedral configurations (M) and (N) (Fig. 3).

The PQS model calculations for these structures were performed as follows:

(I)

$$V_{kk}=V_{zz}=4\{Cl\}^{tba}-2\{Me\}^{tbe}-\{CH_2\}^{tbe}$$

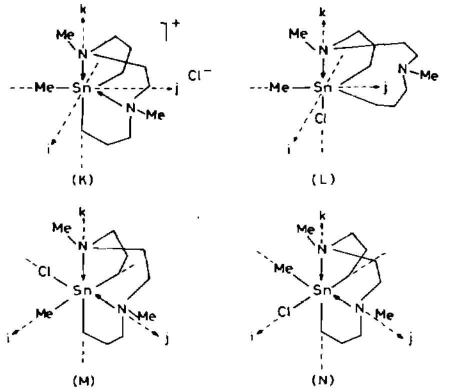

Fig. 3. Principal EFG axes for the structural alternatives (K)–(N) of $Me(Cl)Sn[CH_2CH_2CH_2N(Me)CH_2]_2$.

$$V_{jj}=V_{xx}=2\{CH_2\}^{tbe}-0.5\{Me\}^{tbe}-2\{Cl\}^{tba}$$

$$V_{ii}=V_{yy}=2.5\{Me\}^{tbe}-\{CH_2\}^{tbe}-2\{Cl\}^{tba}$$

$$\eta=(3\{Me\}^{tbe}-3\{CH_2\}^{tbe})/V_{zz}$$

(J)

$$V_{kk}=V_{zz}=2\{PPh_2\}^{tba}+2\{Cl\}^{tba}-2\{Me\}^{tbe}+\{CH_2\}^{tbe}$$

$$V_{jj}=V_{xx}=2\{CH_2\}^{tbe}-0.5\{Me\}^{tbe}-\{PPh_2\}^{tba}-\{Cl\}^{tba}$$

$$V_{ii}=V_{yy}=2.5\{Me\}^{tbe}-0.5\{CH_2\}^{tbe}-\{PPh_2\}^{tba}-\{Cl\}^{tba}$$

$$\eta=(3\{Me\}^{tbe}-3\{CH_2\}^{tbe})/V_{zz}$$

(K)

$$V_{jj}=V_{zz}=2\{Me\}^{tbe}-2\{N\}^{tba}-\{CH_2\}^{tba}$$
$$-0.25\{CH_2\}^{tbe}-0.25\{N\}^{tbe}$$

$$V_{ii}=V_{xx}=-\{Me\}^{tbe}-\{N\}^{tba}-\{CH_2\}^{tba}$$
$$+1.25\{CH_2\}^{tbe}+1.25\{N\}^{tbe}$$

$$V_{kk}=V_{yy}=2\{Me\}^{tbe}-\{N\}^{tba}-\{CH_2\}^{tba}$$
$$-0.25\{CH_2\}^{tbe}+0.25\{N\}^{tbe}$$

$$\eta=(-3\{Me\}^{tbe}+1.5\{CH_2\}^{tbe}+1.5\{N\}^{tbe})/V_{zz}$$

(L)

$$V_{kk}=V_{zz}=2\{N\}^{tba}+2\{Cl\}^{tba}-\{Me\}^{tbe}-2\{CH_2\}^{tbe}$$

$$V_{ii}=V_{xx}-\{Me\}^{tbe}-\{Cl\}^{tba}-\{N\}^{tba}+2.5\{CH_2\}^{tbe}$$

$$V_{jj}=V_{yy}=2\{Me\}^{tbe}-\{N\}^{tba}-\{Cl\}^{tba}-0.5\{CH_2\}^{tbe}$$

$$\eta=0$$

(M)

$$V_{kk}=V_{yy}=\{N\}+\{CH_2\}-\{Cl\}-\{Me\}$$

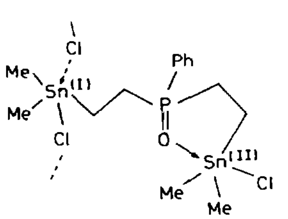

Fig. 4. Schematic representation of the molecular structure of $Ph(O)P(CH_2CH_2SnMe_2Cl)_2$.

$$V_{ii}=V_{zz}=2\{Me\}+\{CH_2\}-\{Cl\}-2\{N\}$$

$$V_{jj}=V_{xx}=2\{Cl\}+\{N\}-\{Me\}-2\{CH_2\}$$

$$\eta=\{Me\}+2\{CH_2\}-3\{Cl\})/V_{zz}$$

(N)

$$V_{kk}=V_{yy}=\{N\}+\{CH_2\}-\{Cl\}-\{Me\}$$

$$V_{ii}=V_{zz}=2\{Cl\}+\{CH_2\}-2\{N\}-\{Me\}$$

$$V_{jj}=V_{xx}=2\{Me\}+\{N\}-2\{CH_2\}-\{Cl\}$$

$$\eta=0$$

The calculations suggest the structure (L) ($QS_{calc}=-3.41$ mm/s, $\eta=0$) or (M) ($QS_{calc}=+3.58$ mm/s, $\eta=0.99$) for compound **16**, but unambiguously exclude the arrangements (K) ($QS_{calc}=+1.17$ mm/s, $\eta=0.65$) and (N) ($QS_{calc}=+0.02$ mm/s, $\eta=0$).

This is in contradiction with our interpretation of the results of temperature-dependent NMR studies [15]. Unfortunately, we were not able to establish the molecular structure of this interesting compound because the single crystals decomposed under X-ray irradiation.

Finally, a comparison of the crystal structure [16] and the Mössbauer data on compound **25**, $(Me_2ClSnCH_2CH_2)_2P(O)Ph$, illustrates the limitations of Mössbauer spectroscopy. The X-ray crystal determination reveals for **25** a monocyclic structure with one intramolecularly pentacoordinated tin atom and the other one intermolecularly bridged to a second molecule via a Sn...Cl interaction (Fig. 4). The two tin centres exhibit significantly different deviations from the ideal trigonal bipyramidal geometry. Actually, the Mössbauer data (*IS* 1.36; *QS* 3.30 mm/s) confirm the pentacoordination of the tin atoms, but they cannot distinguish between the two different ligand polyhedra, for which different *QS* values of −3.39 (Sn(I)) and −3.63 (Sn(II)) mm/s were calculated. A similar effect has already been observed for $(Ph_2ClSn)_2CH_2\cdot HMPA$ [17].

Experimental

The syntheses of compounds **1**, **2** [18], **3–7** [11, 18], **8–10** [12, 15], **11–16** [12, 19], **17** [19], **18**, **19**

[10], **20–24** [20–22] and **25** [16] have been described elsewhere.

The Mössbauer spectra were recorded at liquid nitrogen temperature. The Mössbauer parameters were derived by computer evaluation of the spectra. The *IS* values are referred to that of SnO_2. The reproducibility of the Mössbauer parameters was found to be better than +0.03 mm/s.

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