Molecular structure and stereoisomerization of bis(2-dimethylchlorostannylethyl)phenylphosphine oxide

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Abstract

NMR data (^{1}H, ^{13}C, ^{119}Sn, ^{31}P) have revealed that in donor-free solvents bis(2-dimethylchlorostannylethyl)phenylphosphine oxide (1) has a symmetric bicyclic structure with the two tin atoms intramolecularly coordinated to the P=O group. In the presence of pyridine one intramolecular P=O → Sn coordination is broken and the fifth coordination site of the corresponding tin atom occupied by a solvent molecule. In both solvents compound 1 undergoes various ligand exchange processes.

The crystal structure of compound 1 has been determined by X-ray analysis and refined to a final R value of 0.047. The compound crystallizes in the monoclinic space group $P2_1/c$. The structure consists of monocyclic units involving an intramolecular P=O → Sn coordination which are associated through intermolecular Sn–Cl...Sn bridges to give infinite zigzag chains parallel to the *b*-axis. The ligand polyhedra of the two tin atoms exhibit trigonal-bipyramidal geometry, with different deviations from the ideal trigonal-bipyramidal arrangement.

Introduction

The structural feature of functionally-substituted triorganotin halides containing a donor center appropriately located in the functional substituent is characterized by an intramolecular donor-tin coordination leading to a cyclic structure in which the five-coordinated tin atom usually has a trigonal-bipyramidal geometry [1,2,3]. The presence of two halogenated tin atoms in such molecules gives rise to further structural modifications involving the possibility of the formation of tin halogen bridges.

Thus, the structure of 3,3-bis(dimethylhalostannyl)propyl ethyl sulfoxides (Me$_2$XSn)$_2$CH CH$_2$CH$_2$S(O)Et (X = Cl, Br), both in the solid state and in solution, consists of monomeric units with non-equivalent pentacoordinated tin atoms. One

tin center achieves the five-coordinated state by intramolecular coordination to the sulfoxide group, and the other one by formation of an intramolecular tin halogen bridge [3]. In contrast, bis(3-dimethylchlorostannylpropyl)sulfide ($Me_2ClSnCH_2$-$CH_2CH_2)_2S$ has a symmetric bicyclic structure in the solid state, with the sulfur atom coordinated to the two tin atoms without any intra- or intermolecular tin-chlorine interaction [4]. In connection with our studies on organophosphorus-substituted organotin compounds we have prepared bisfunctional compounds of the type $[Me_3Sn(CH_2)_n]_2P(O)R$ ($n = 2$, 3; R = Ph, t-Bu) and their bis-halogenated derivatives $[Me_2XSn(CH_2)_n]P(O)R$, and we discuss here the stereochemistry and dynamic behaviour of bis(2-dimethylchlorostannylethyl)phenylphosphine oxide ($Me_2ClSnCH_2CH_2)_2P(O)Ph$ on the basis of its crystal structure and NMR data.

Results and discussion

Preparation and spectral properties

Bis(2-dimethylchlorostannylethyl)phenylphosphine oxide (**1**) was prepared by chlorination of bis(2-trimethylstannylethyl)phenylphosphine oxide with hydrogen chloride in ether. Osmometry in $CHCl_3$ indicates that **1** is monomeric. The shift of the P=O stretching frequency in the IR spectra of **1** in both the solid state and $CHCl_3$ solution (KBr: 1140 cm^{-1}, $CHCl_3$: 1145 cm^{-1}) by about 50 cm^{-1} to lower values, comparable to that for $(Me_3SnCH_2CH_2)_2P(O)Ph$ (film: 1195 cm^{-1}), indicates P=O $\rightarrow$ Sn coordination.

The NMR parameters (^1H, ^{13}C, ^{119}Sn, ^{31}P) of **1** and two related compounds in various solvents at ambient and low temperature are listed in Table 1. Taking into account our previous results [1,2,5–8], the following conclusions can be drawn about the stereochemistry of **1**.

(i) In donor-free solution both the ^{119}Sn and ^{31}P NMR spectra of **1** show one signal with only one $J(^{119}Sn,^{31}P)$ coupling constant, which is relatively little changed by temperature variation. The ^{31}P-signal is shifted to lower field and the ^{119}Sn signal by about 100 ppm to higher field compared with values for $(Me_3SnCH_2CH_2)_2P(O)Ph$ and Me_3SnCl ($\delta(^{119}Sn)$ (C_6D_6) 164.2 ppm [9]), respectively. In the light of the value of $J(^{119}Sn,^{31}P)$, which represents the sum of $^3J(^{119}SnCC^{31}P)$ and $^2J(^{119}SnO^{31}P)$ [5–8], and the monomeric structure of the compound, these results show that **1** in donor-free solvents up to 185 K has a bicyclic structure with the two tin centers coordinated to the phosphine oxygen lone pairs (Fig. 1a). The ^1H and ^{13}C NMR data of **1**, particularly those of the tin-methyl groups, support the suggested structure.

(ii) At sufficiently low temperatures the single ^1H and ^{13}C NMR absorption of the tin-methyl groups splits into two lines, indicating an inter- or intramolecular exchange process.

(iii) In the presence of pyridine the ^{119}Sn NMR signal of **1** is shifted to higher field and the $J(^{119}Sn,^{31}P)$ coupling constant markedly increased. Furthermore, the ^{119}Sn NMR singlet at ambient temperature is split into two signals of same intensity at lower temperature, indicating that the tin atoms in **1** are non-equivalent under slow exchange conditions. The observed coupling constant of 282 Hz corresponds to a $^3J(SnCCP)$ coupling [6], and allows the assignment of the signal at -37.8 ppm to a tin atom intermolecularly coordinated to pyridine (Fig. 1b). The other tin atom (δ -0.5 ppm) is intramolecularly coordinated, but the $J(Sn,P)$ ring coupling (expected

Table 1

Selected NMR parameters (^{1}H, ^{13}C, ^{119}Sn, ^{31}P) of **1** and related compounds

	Temper-ature (K)	Solvent	Nucleus studied	δ (ppm)	J (Hz)	
^{1}H	298	CD$_2$Cl$_2$	SnCH_3	0.67	^2J(SnCH)	65.6
	188	CD$_2$Cl$_2$	SnCH_3	0.72(1), 0.58(2)	^2J(SnCH)	67.1(1), 62.3(2)
	298	CD$_2$Cl$_2$/Pyr-d_5	SnCH_3	0.70	^2J(SnCH)	67.1
	178	CD$_2$Cl$_2$/Pyr-d_5	SnCH_3	0.75, 0.65, 0.62, 0.46	^2J(SnCH)	n.o. [a]
^{13}C	302	CD$_2$Cl$_2$	SnCH_3	1.1	^1J(SnC)	460.7
			SnCH_2	9.7	^1J(SnC)	472.3; ^2J(CCP) 5.4
			PCH_2	24.8	^2J(CCSn)	17.9; ^1J(CP) 65.9
	203	CD$_2$Cl$_2$	SnCH_3	2.4(1), 0.8(2)	^1J(SnC)	n.o.
			SnCH_2	9.7	^1J(SnC)	480.0; ^2J(CCP) n.o.
			PCH_2	23.8	^2J(CCSn)	n.o.; ^1J(CP) 66.0
^{119}Sn	303	Toluene-d_8		60.5	J(Sn, P)	67.1
	183	Toluene-d_8		59.2	J(Sn, P)	66.2
	303	Tol-d_8/Pyr-d_5		7.0	J(Sn, P)	168.0
	183	Tol-d_8/Pyr-d_5		−0.5(1); −37.8(2)	J(Sn, P) ^3J(SnCCP)	n.o.(1), 282(2)
^{31}P	303	CDCl$_3$		58.0	J(Sn, P)	66.6
	303	CDCl$_3$/Pyr-d_5		56.2	J(Sn, P)	163.8
	213	Tol-d_8/Pyr-d_5		60.8	J(Sn, P)	181.0
(Me$_3$SnCH$_2$-CH$_2$)$_2$P(O)Ph						
^{31}P	303	CDCl$_3$		44.0	^3J(SnCCP)	207.9
^{119}Sn	303	CDCl$_3$		10.7	^3J(SnCCP)	206.8
Me$_2$Sn(Cl)CH$_2$-CH$_2$P(O)PhBu-t [7]						
^{31}P	303	CDCl$_3$		60.0	J(Sn, P)	38.8
^{119}Sn	303	CDCl$_3$		2.9	J(Sn, P)	36.6

[a] n.o. not observed.

to have a value of about 40 Hz [6], see Me$_2$ClSnCH$_2$CH$_2$P(O)PhBu-t) at low temperature is not observable because of the broadening of the signals.

(iv) The conclusions in (iii) are confirmed by the ^{1}H NMR spectra in the presence of pyridine. The single tin-methyl absorption at ambient temperature is split into four different signals under slow exchange conditions, indicating the
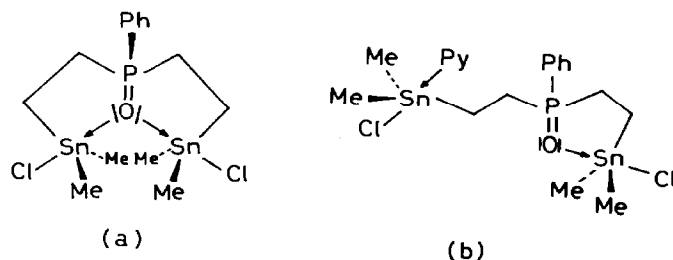

Fig. 1. Proposed structure of **1** in donorfree solvents (a) and in pyridine (b).

Table 2

Dynamic NMR parameters of **1**

Solvent	Concentration (mol/l)	Nucleus studied	Coalescence temperature (K)	$\Delta\nu$ (Hz)	$G_{coal.}^{*}$ (kJ mol^{-1})
CD$_2$Cl$_2$	0.018	SnCH_3	255	25.7	53.7
CD$_2$Cl$_2$	0.18	SnCH_3	210	26.0	43.7
CD$_2$Cl$_2$/pyr-d_5	0.18	SnCH_3	191	50.3	38.5

non-equivalence of the four methyl groups arising from the different coordination modes at the two tin centres.

(v) The dynamic process in donor-free solution is concentration-dependent (parameters see Table 2), but the proved monomeric structure of **1** excludes association processes as the reason for the observed NMR line shape variations.

(vi) The NMR data (Table 2) permit the following suggestions to be made about the dynamic behaviour of **1**. The mode of the process is solvent-dependent. In the ground state **1** has a bicyclic structure. The two tin centers are pentacoordinated, and there are two types of methyl groups, differing in respect of their position relative to the *P*-phenyl group. In a donor-free solvent the tin atoms are attacked by the chlorine atoms of a second molecule of **1** to induce a stereoisomerization of the tin atom via a hexacoordinated transition state with intermolecular tin-chlorine bridges [7,10,11]. Increase in the concentration of the solution favours this dynamic process, lowering the transition state energy by facilitating the chlorine-tin exchange. The near constancy of the tin-phosphorus coupling constant on temperature and concentration variations indicates that the bicyclic structure is maintained during this process.

However, the presence of pyridine gives rise to two different dynamic processes. On the one hand the donor solvent breaks one intramolecular P=O → Sn link to induce a coordination exchange between intramolecularly (involving the P=O group) and intermolecularly (involving a pyridine molecule) five-coordinated tin atoms. In addition every tin center undergoes the stereoisomerization process discussed above, involving a hexacoordinated transition state with the donor solvent as the sixth ligand.

Crystal and molecular structure

Positional and thermal parameters for compound **1** are listed in Table 3. The molecular structure with the atom numbering is shown in Fig. 2, and the molecular packing is shown in Fig. 3. Selected bond lengths and angles are given in Table 4, and torsion angles defining the chelate ring conformation in Table 5.

The most striking feature of the molecular structure is the marked non-equivalence of the two tin atoms. In contrast to its symmetric bicyclic structure in donor-free solvents, in the solid state compound **1** consists of monocyclic structural units with only one tin atom intramolecularly coordinated to the P=O group, with the other intermolecularly linked to a neighbouring molecule via a bridging Sn...Cl interaction. The geometry around each tin atom is that of a distorted trigonal bipyramid. In both cases the equatorial plane is occupied by three carbon atoms, belonging to the methyl groups and the P-bonded ethylene groups, and one axial

Table 3

Final fractional coordinates and equivalent isotropic thermal parameters (Å^2).

$$U_{eq} = 1/3 \sum_i \sum_j U_{ij} a_i^\star a_j^\star (a_i \cdot a_j)$$

Atom	x	y	z	U_{eq}
Sn(1)	0.26086(5)	−0.02873(4)	0.53299(6)	0.0549(3)
Sn(2)	0.34698(5)	0.38647(4)	0.57842(5)	0.0493(3)
Cl(1)	0.0807(2)	−0.0295(2)	0.3355(2)	0.079(1)
Cl(2)	0.4886(2)	0.4991(2)	0.7042(3)	0.079(1)
P	0.1552(2)	0.2447(1)	0.5657(2)	0.043(1)
O	0.2024(5)	0.2848(3)	0.4784(5)	0.046(3)
C(1)	0.0051(7)	0.2800(5)	0.5370(7)	0.048(4)
C(2)	−0.0511(9)	0.2540(6)	0.6129(9)	0.069(5)
C(3)	−0.1677(10)	0.2803(7)	0.5863(13)	0.089(7)
C(4)	−0.2286(10)	0.3276(8)	0.4841(14)	0.097(7)
C(5)	−0.1740(10)	0.3536(8)	0.4094(12)	0.095(7)
C(6)	−0.0546(8)	0.3300(6)	0.4360(9)	0.066(5)
C(7)	0.1489(7)	0.1362(5)	0.5477(8)	0.053(4)
C(8)	0.2714(7)	0.0994(5)	0.5638(9)	0.059(4)
C(9)	0.2036(9)	−0.0982(6)	0.6503(9)	0.071(5)
C(10)	0.3840(9)	−0.0842(7)	0.4706(11)	0.082(6)
C(11)	0.2553(8)	0.2723(6)	0.7263(7)	0.060(4)
C(12)	0.2886(8)	0.3616(6)	0.7263(8)	0.064(4)
C(13)	0.4852(9)	0.3100(8)	0.5723(13)	0.100(6)
C(14)	0.2527(11)	0.4569(8)	0.4166(10)	0.095(6)

Cl(2′): Symmetry code $1 - x$, $y - 0.5$, $1.5 - z$

position is occupied by a chlorine atom. But the pentacoordination is achieved in quite different ways: the second axial position at Sn(2) is occupied by the oxygen atom of the P=O group, whereas the coordination sphere around Sn(1) is completed
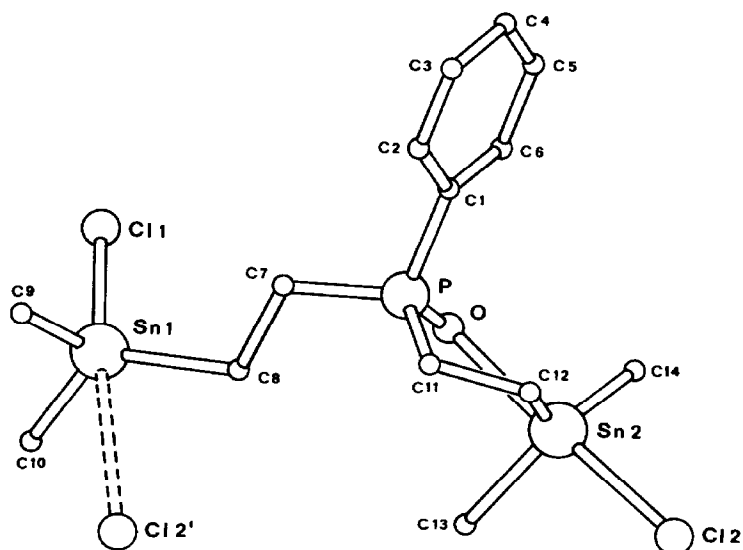

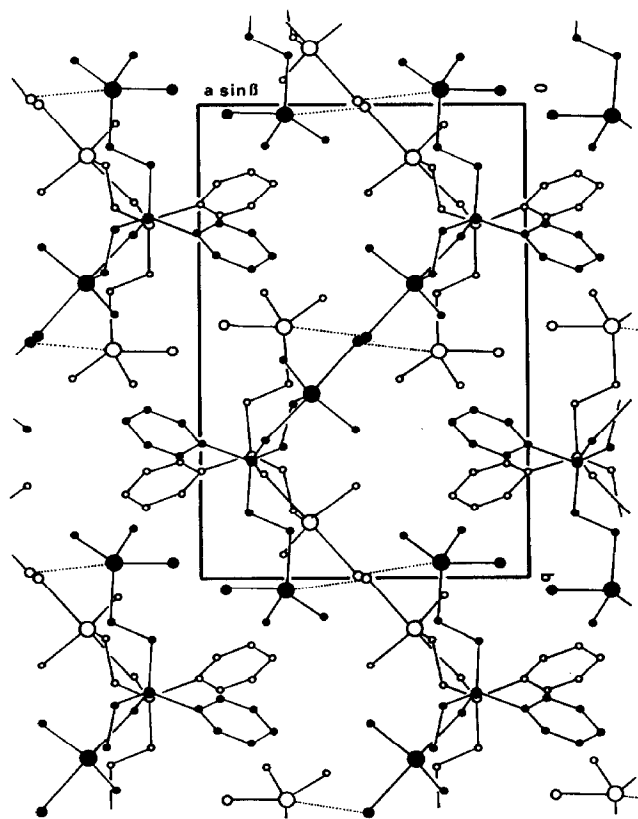Fig. 2. Molecular structure of **1** with the atom-numbering scheme.

Fig. 3. Crystal structure of **1** projected along |001|.

by the chlorine atom Cl(2′) of a neighbouring molecule, which is related to the original molecule by the space group two-fold screw axis. The molecules are associated through the Sn–Cl...Sn bridges into infinite zigzag chains parallel to the *b*-axis (Fig. 3).

Compared with that of Sn(2), the trigonal-bipyramidal skeleton of Sn(1) is more distorted towards a monocapped tetrahedron owing to the lower donor strength of the bridging chlorine atom Cl(2′). This is indicated by the smaller difference between the sums of the equatorial and axial angles at Sn(1) (Δ 53.9°) and Sn(2) (Δ 74°) [12], and the greater displacement of Sn(1) out of the equatorial plane defined by the three carbon atoms (Sn(1): 0.346(1) Å, Sn(2): 0.172(1) Å).

Both the Sn–C bond lengths and the Sn(2)–O distance are in the range observed for other comparable pentacoordinated methyltin compounds [1–3,7]. The difference of 0.121 Å between the bond lengths Sn(1)–Cl(1) and Sn(2)–Cl(2) reflects the shortening of axial Sn–Cl bonds in a trigonal-bipyramidal ligand polyhedron, with increasing deviation from the ideal geometry towards a monocapped tetrahedron. The intermolecular Sn(1)...Cl(2′) distance is considerably shorter than the sum of corresponding Van der Waals radii (3.85 Å [13]) and indicates substantial bonding.

As in various intramolecular pentacoordinated triorganotin halides with a five-membered ring [1–3,7,10], the torsion angles in Table 5 indicate that **1** has an envelope conformation of the chelate ring. The C(12), Sn(2), O and P atoms are nearly coplanar, and the ring is puckered at the C(11) edge.

Table 4

Selected bond lenghts (Å) and angles (°)

Sn(1)–Cl(1)	2.415(3)	Cl(1)–Sn(1)–C(8)	97.5(3)
Sn(1)–C(8)	2.136(9)	Cl(1)–Sn(1)–C(9)	101.2(3)
Sn(1)–C(9)	2.098(11)	Cl(1)–Sn(1)–C(10)	99.5(3)
Sn(1)–C(10)	2.114(12)	C(9)–Sn(1)–C(8)	116.4(4)
Sn(1)…Cl(2′)	3.314(3)	C(9)–Sn(1)–C(10)	116.9(4)
		C(8)–Sn(1)–C(10)	118.8(4)
		Cl(1)–Sn(1)…Cl(2′)	172.10(7)
Sn(2)–Cl(2)	2.536(3)	Cl(2)–Sn(2)–C(12)	93.0(3)
Sn(2)–C(12)	2.139(10)	Cl(2)–Sn(2)–C(13)	95.6(3)
Sn(2)–C(13)	2.120(12)	Cl(2)–Sn(2)–C(14)	95.4(3)
Sn(2)–C(14)	2.090(13)	C(12)–Sn(2)–C(13)	116.1(5)
Sn(2)–O	2.352(5)	C(12)–Sn(2)–C(14)	125.4(4)
		C(13)–Sn(2)–C(14)	116.6(5)
		Cl(2)–Sn(2)–O	172.7(2)
P–O	1.501(6)	C(1)–P–C(7)	107.9(4)
P–C(11)	1.802(9)	C(1)–P–C(11)	107.7(4)
P–C(7)	1.797(9)	C(1)–P–O	111.7(4)
C(7)–C(8)	1.542(12)	Sn(2)–C(12)–C(11)	111.1(6)
C(11)–C(12)	1.526(14)	C(12)–C(11)–P	108.0(6)
		C(11)–P–O	107.3(4)
		P–O–Sn(2)	113.4(3)
		O–Sn(2)–C(12)	80.2(3)

Table 5

Endocyclic torsion angles (°) of the five-membered chelate ring

Sn(2)–C(12)–C(11)–P	− 49.3(7)
C(12)–C(11)–P–O	+ 40.5(7)
C(11)–P–O–Sn(2)	− 14.1(4)
P–O–Sn(2)–C(12)	− 8.3(4)
O–Sn(2)–C(12)–C(11)	+ 33.2(6)

Finally, the Mössbauer data (IS: 1.36 mm s^{-1} (relative to SnO$_2$); QS: 3.30 mm s^{-1}) confirm the existence of pentacoordinated tin in **1**, but the insensitivity of the method does not allow distinction between the two different tin centers.

Experimental

All manipulations were carried out under dry argon. The NMR spectra were recorded with a Bruker WP 200 spectrometer, ^{13}C at 50.39 MHz, ^{31}P at 81.026 MHz, and ^{119}Sn at 74.64 MHz. References were TMS (^{1}H, ^{13}C), 85% H$_3$PO$_4$ (^{31}P), and Me$_4$Sn (^{119}Sn). Low-field shifts are positive.

Infrared spectra were obtained with a Specord 71 instrument (Carl Zeiss Jena).

Materials

Bis(trimethylstannylethyl)phenylphosphine oxide. (a) A mixture of 6.6 g (40 mmol) of divinylphenylphosphine [14] and 13.2 g (80 mmol) of trimethyltin hydride was slowly warmed to 70°C during 10 h with portion-wise addition of 3 mol% of AIBN.

The mixture was then distilled in vacuum, to give 16.4 g (83%) of $(Me_3SnCH_2CH_2)_2PPh$; b.p. 130–132°C/0.04 Torr. Anal.: Found: C, 38.85; H, 6.24. $C_{16}H_{31}PSn_2$ calcd.: C, 39.08; H, 6.35%. NMR: ^1H (C_6D_6) δ 0.08 (s, 18H, $SnCH_3$), $^2J(SnCH)$ 52.5 Hz. ^{119}Sn (C_6D_6) δ 3.3 (d) ppm, $^3J(SnCCP)$ 136.2 Hz. $^{31}P(C_6D_6)$ δ −12.3 (s) ppm, $^3J(PCCSn)$ 144.8 Hz.

(b) Oxidation of $(Me_3SnCH_2CH_2)_2PPh$ to its oxide was performed in acetone at room temperature by dropwise addition of a saturated solution of $KMnO_4$ in the same solvent. The reaction was complete when the solution over the precipitate of MnO_2 became violet. The mixture was filtered and the filtrate distilled; b.p. 144–147°C/0.07 Torr. Anal.: Found: C, 38.00; H, 5.99. $C_{16}H_{31}OPSn_2$ calcd.: C, 37.85; H, 6.15%. IR (film) 1195(s) (P=O) cm^{-1}. ^1H NMR ($CDCl_3$) δ 0.10 (s, 18H, $SnCH_3$), $^2J(SnCH)$ 53.6 Hz.

Bis(dimethylchlorostannylethyl)phenylphosphine oxide (1). A solution of 1.6 g (44 mmol) of hydrogen chloride in ether was added dropwise at −30°C to 10.6 g (21 mmol) of $(Me_3SnCH_2CH_2)_2P(O)Ph$ in $CHCl_3$. The mixture was then allowed to warm to room temperature, and after some time the solvent was removed to leave a viscous residue, which later crystallized. Recrystallization from CH_2Cl_2/hexane gave 7.7 g (70%) 1; m.p. 148–150°C. Anal.: Found: C, 30.43; H, 4.55; Cl, 12.76. $C_{14}H_{25}OPCl_2Sn_2$ calcd.: C, 30.66; H, 4.59; Cl, 12.93%. IR (KBr) 1140(s), ($CHCl_3$) 1145(s) (P=O) cm^{-1}.

X-ray structure determination

Preliminary unit cell dimensions and space group $P2_1/c$ were determined from oscillation and Weissenberg photographs. All other operations were carried out on an Enraf–Nonius CAD-4 diffractometer using graphite-monochromatized Mo-K_α radiation (λ 0.71069 Å). Accurate cell parameters were obtained by a least-squares fit of the setting angles of 25 reflections.

Crystal data: $C_{14}H_{25}OPCl_2Sn_2$, $M = 548.7$, monoclinic, space group $P2_1/c$, a 12.108(3), b 16.479(5), c 11.535(6) Å, β 114.46(3)°, V 2095.0 Å^3, $Z = 4$, D_c 1.74 g cm^{-3}, $F(000) = 1032$.

Intensity data for 4270 unique reflections ($1.5° < \theta < 27°$) were measured and corrected for Lorentz and polarization effects but not absorption and extinction (μ(Mo-K_α) 25.1 cm^{-1}, crystal size 0.25 × 0.35 × 0.38 mm). The structure was solved by the heavy atom method. The coordinates of the Sn atoms were deduced from a Patterson map and two subsequent Fourier syntheses revealed the positions of all non-H atoms. Their positional and anisotropic thermal parameters were refined by least-squares methods, but the H atoms positions were placed in calculated positions and not refined. For the refinement 3855 reflections with $| F_0 | \geqslant 3.92\sigma(F_0)$ were used. Seven reflections with low indices and high intensity exhibiting large $\Delta F/\sigma(F)$ were apparently affected by extinction, and therefore omitted from the final least-squares cycles. The final R-value was 0.047.

All calculations were carried out on an ESER 1040 computer of the Halle University with the programs SHELX-76 [15], GEOME [16] and PLANE [17].

Acknowledgement

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