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SINGLE CRYSTAL AND MOLECULAR STRUCTURE OF METHYLPHENYLTIN(IV) DICHLORIDE, INTERMEDIATE BETWEEN POLYMERIC AND MONOMERIC R_2SnCl_2 STRUCTURES

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Summary

Methylphenyltin(IV) dichloride crystallizes with a roughly linear array of four tin atoms, each tetrahedrally coordinated to two chlorine atoms, as well as to a methyl and a phenyl group. Each successive pair of tin atoms appears incipiently bridged by two chlorine atoms, there being tin to neighboring chloride distances of 3.4–3.8 Å which, if shortened, would leave each successive pair of tin atoms doubly bridged by chlorine atoms. The structure is thus reminiscent of the one dimensional polymers formed by R_2SnCl_2 (R = Me, Et).

Introduction

Diorganotin(IV) dichlorides, R_2SnCl_2, have displayed a variety of structural motifs in the solid state [1,2] X-ray diffraction studies have yielded structural parameters for six members of this family [3–10]. Categorization of the structural types exhibited by these six materials depends upon differentiation between Sn–Cl distances to be considered as bonding, those to be considered as weakly bonding or interactive and those to be viewed as of no significance. Distances *d*(Sn–Cl) of 2.40 Å or less may clearly be viewed as bonding, and all six structures display two of these Sn–Cl distances per tin atom. These six structures also display tin–chloride distances of 3.21–3.976(1) Å, and it is these distances which are at least 0.8 Å greater than those of a clearly bonding nature which have been the subject of interpretation. Distances *d*(Sn–Cl) of 2.805, 2.96 and 2.78 Å have been observed as bridging tin distances in the transition-metal complexes $(bipy)Mo(CO)_3(SnMeCl_2)Cl$ [11], $(dth)W(CO)_3(SnMeCl_2)Cl$ [12] and $(dth)Mo(CO)_3(SnCl_3)Cl$ [13], respectively

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(bipy = α,α'-bipyridyl, dth = 2,5-dithiahexane). There is some disagreement as to the van der Waals radius of tin [14]. Application of a commonly quoted value of 3.85 Å for the sum of van der Waals radii [14] of Sn and Cl as an upper limit for interactive distances allows the assignment of the six structures to five of the possible structural categories for R_2SnCl_2:

A. Monomer (R = bis(biphenylyl-2)] [3], (η^5-$C_5H_5FeC_5H_4$-η^5 [4]) (4)
B. Oligomer
  1. Dimer
  2. Trimer
  3. Tetramer (R = C_6H_5) [5,6] (4,6)
  4. Higher
C. 1-Dimensional polymer
  1. Singly-bridged (R = C_6H_{11}) [7,8] (5)
  2. Doubly-bridged
    a. Chelated
      (i) isobidentate (R = $ClCH_2$) [6] (8)
      (ii) anisobidentate
    b. Symmetrically bridged
    c. Asymmetrically bridged (R = CH_3 [9], C_2H_5 [10]) (6) (6)
D. 2-Dimensional polymer

The numbers in parentheses indicate the coordination number for tin when the limiting value of 3.85 Å for Sn–Cl coordination is imposed.

The issue of tin coordination is not entirely settled by this categorization. Along with the expectation of an internuclear separation of less than the sum of the van der Waals radii, goes the expectation of distortion of the tetrahedral bond angles about tin with the addition of more bonding atoms. Tin coordination numbers range from 4–8 in the above listing. It is not clear what the non-distorted angles for an R_2SnCl_2 monomer should be. Dimethyltin dichloride was reported to display tetrahedral Cl–Sn–Cl angles of 110(5)° in early gas-phase experiments [15], however, later workers have suggested that this is not reliable [5]. The six structures as a group display Cl–Sn–Cl angles which range from 93(2)–100° and C–Sn–C angles in the range 123.5(4)–135(6)°, narrow ranges considering the errors and the range of coordination numbers displayed. The bis(ferrocenyl)- [4] and bis(diphenylyl-2)tin dichloride [3] structures, which are clearly monomeric, display Cl–Sn–Cl and C–Sn–C angles of 98.8(2) and 120.4(6)° [4] and 93(2) and 124(4)° [3], respectively, which are at the limits of either range. However, both angles should be subject to the influence of the steric bulk of the R group, and the ferrocenyl and diphenylyl-2 groups are bulky ligands. It seemed that studying an example in which the two organic groups were not the same might yield a novel result. We report here the single crystal X-ray structure of methylphenyltin(IV) dichloride [16–31], $CH_3(C_6H_5)SnCl_2$, which may be interpreted in light of previous work.

Results and discussion

Crystal and data collection parameters are listed in Table 1, atomic coordinates for all non-hydrogen atoms in Table 2, anisotropic thermal parameters in Table 3 and internuclear distances and angles in Table 4. Structure factors are available in the supplementary materials. The molecular geometry and labeling scheme are given in Fig. 1.

TABLE 1

CRYSTAL DATA FOR $CH_3(C_6H_5)SnCl_2$ [a]

Formula	$C_7H_8SnCl_2$
Mol. wt.	281.74
a	17.312(8) Å
b	6.704(1)
c	17.384(11)
α	90.0
β	101.38(4)°
γ	90.0
V	1977.5(15) Å^3
$F(000)$	1072
μ(Mo-K_α)	30.72 cm^{-1}
λ(Mo-K_α)	0.71069 Å
D_{calc}	1.89 g cm^{-3}
Z	8
Obs. refl.	1793
R/R_w	8.5/13.1
Space group	$P2_1/a$

[a] Estimated standard deviations in parentheses.

The refinement of the phenyl rings proceeded with considerable difficulty as is evident from the values of the anisotropic thermal parameters for the carbon atoms and from the bond angles and distances calculated from the final positional parameters. The refinement difficulties are not unexpected in view of the low melting point of the material and the fragile nature of the crystals. However, the tin

TABLE 2

POSITIONAL PARAMETERS FOR METHYLPHENYLTIN(IV) DICHLORIDE

	x ($\sigma(x)$)	y ($\sigma(y)$)	z ($\sigma(z)$)
Sn(1)	0.4826(1)	0.1118(4)	0.3731(1)
Sn(2)	0.3887(1)	0.1090(4)	0.0987(1)
Cl(1)	0.4532(5)	0.3962(15)	0.2894(6)
Cl(2)	0.4678(5)	0.2593(12)	0.4910(5)
Cl(3)	0.4906(6)	−0.0702(16)	0.1800(6)
Cl(4)	0.3752(7)	−0.0917(18)	−0.0146(6)
C(1)	0.602(2)	0.074(4)	0.386(2)
C(2)	0.645(2)	0.240(6)	0.430(6)
C(3)	0.741(2)	0.196(7)	0.418(2)
C(4)	0.765(2)	0.030(6)	0.395(2)
C(5)	0.716(1)	−0.127(5)	0.362(2)
C(6)	0.639(3)	−0.099(6)	0.355(2)
C(7)	0.383(2)	−0.098(5)	0.348(2)
C(8)	0.285(3)	0.071(6)	0.144(3)
C(9)	0.238(2)	−0.116(7)	0.139(2)
C(10)	0.180(2)	−0.158(8)	0.151(3)
C(11)	0.128(3)	0.000(8)	0.211(3)
C(12)	0.185(3)	0.182(7)	0.209(2)
C(13)	0.251(2)	0.227(6)	0.188(2)
C(14)	0.444(2)	0.393(6)	0.059(2)

TABLE 3

SELECTED BOND ANGLES (°) AND DISTANCES (Å) FOR METHYLPHENYLTIN(IV) DICHLORIDE

Sn(1)-Cl(1)	2.39(1)	Cl(1)-Sn(1)-Cl(2)	98.6(3)
Sn(1)-Cl(2)	2.335(9)	Cl(1)-Sn(1)-Cl(2′)	173.7(3)
Sn(1)-Cl(2′)	3.422(9)	Cl(1)-Sn(1)-C(1)	104.7(9)
Sn(1)-C(1)	2.05(3)	Cl(1)-Sn(1)-C(7)	109.0(8)
Sn(1)-C(7)	2.20(3)	Cl(2)-Sn(1)-Cl(2′)	76.0(3)
Sn(2)-Cl(1)	3.81(1)	Cl(2)-Sn(1)-C(1)	103.7(9)
Sn(2)-Cl(3)	2.36(1)	Cl(2)-Sn(1)-C(7)	103.1(9)
Sn(2)-Cl(4)	2.36(1)	Cl(2′)-Sn(1)-C(1)	73.9(9)
Sn(2)-C(8)	2.11(6)	Cl(2′)-Sn(1)-C(7)	75.7(8)
Sn(2)-C(14)	2.29(4)	C(1)-Sn(1)-C(7)	133.(1)
		Cl(1)-Sn(2)-Cl(3)	71.3(3)
		Cl(1)-Sn(2)-Cl(4)	168.5(3)
		Cl(1)-Sn(2)-C(8)	82.(1)
		Cl(1)-Sn(2)-C(14)	77.(1)
		Cl(3)-Sn(2)-Cl(4)	98.6(4)
		Cl(3)-Sn(2)-C(8)	108.(1)
		Cl(3)-Sn(2)-C(14)	107.(1)
		Cl(4)-Sn(2)-C(8)	107.(1)
		Cl(4)-Sn(2)-C(14)	102.(1)
		C(8)-Sn(2)-C(14)	130.(2)
		Sn(1)-Cl(1)-Sn(2)	96.7(3)

TABLE 4

COMPARISON DATA FOR DIORGANOTIN(IV) DICHLORIDE STRUCTURES [a]

Compound	Ref.	d(Sn-C) (Å)	d(Sn-Cl) (Å)	Angle C-Sn-C (°)	Angle Cl-Sn - Cl(deg) (°)	d(Sn-Cl) (Å)
$(CH_3)_2SnCl_2$						
X-ray, r.t.	9	2.21	2.40	123.5	93.0	3.54
ed, 85-88 °C	33	2.108	2.327	[b]	107.5	-
$(C_2H_5)_2SnCl_2$		2.132	2.385			3.483
X-ray, r.t.	10	2.167	2.384	134.0	96.0	3.440
$(ClCH_2)_2SnCl_2$						
X-ray, r.t.	6	2.18	2.37	135	97.0	3.71
$(C_6H_{11})_2SnCl_2$			2.371			3.332
X-ray [b]	7	2.151	2.407	134.4	94.3	3.976
X-ray, 25 °C	8	2.156	2.393	131.0	96.8	3.54
		2.159	2.400			
$(C_6H_5)_2SnCl_2$			2.336	123.9		
X-ray, r.t.	5	2.114	2.353	127.0	97.8	3.77
			2.357		101.7	3.78
$(2\text{-}C_6H_5C_6H_4)_2SnCl_2$	3	2.119	2.381	130.8		6.189
X-ray, r.t.		2.140	2.391		99.71	
$(\eta^5\text{-}C_5H_5FeC_5H_4\text{-}\eta^5)_2SnCl_2$	4	2.081	2.350	120.4	99.8	> 6
$CH_3(C_6H_5)SnCl_2$		2.05(3) [c]	2.335(9) [d]			
X-ray, r.t.	[o]	2.11(6) [h]	2.36(1) [i]	130 [e]	98.6(3,4) [f]	3.442(9) [g]
		2.20(3) [k]	2.39(1) [l]	133 [m]		3.56(1) [j]
		2.29(4) [p]				3.81(1) [n]

[a] Estimated standard deviations for the last digit in parentheses. [b] Not given. [c] Sn(1)-phenyl. [d] Sn(1)-Cl(2). [e] Terminal tin atom. [f] Twofold. [g] Sn(1)-Cl(2′). [h] Sn(2)-phenyl. [i] Sn(2)-Cl(3,4). [j] Sn(1)-Cl(3). [k] Sn(1)-methyl. [l] Sn(1)-Cl(1). [m] Interior tin atom. [n] Sn(1)-Cl(1). [o] This work. [p] Sn(2)-methyl.

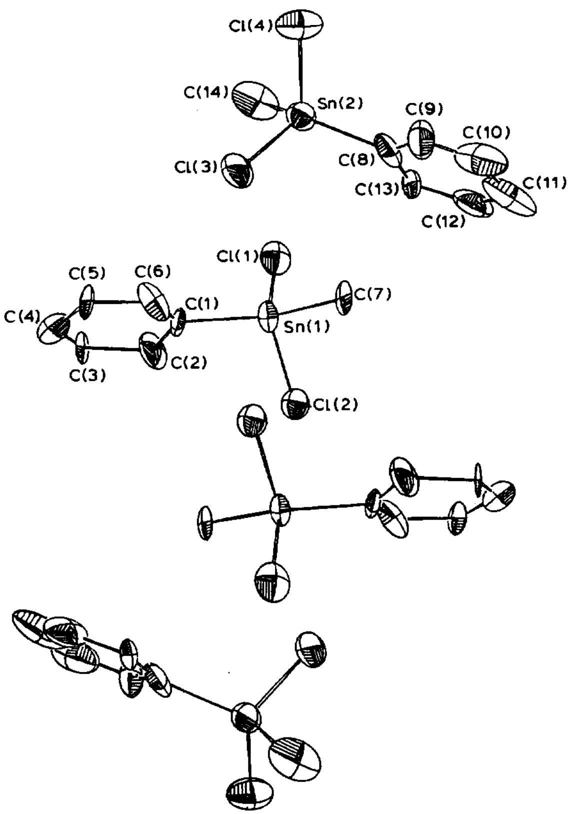

Fig. 1. Methylphenyltin(IV) dichloride, $CH_3(C_6H_5)SnCl_2$, showing the atomic numbering.

and chloride positions are well enough established to permit consideration of the tin bonding.

Methylphenyltin(IV) dichloride, $(C_6H_5)(CH_3)SnCl_2$, crystallizes with two molecules per asymmetric unit. The two tin complexes and two more related to them by the symmetry operation, $1-x$, $-y$, $1-z$, are linked together by a series of Sn–Cl interactions of less than 4 Å. Thus Sn(1) is bound to Cl(1) and Cl(2), but has a distance of 3.42(1) Å to the adjacent Cl(2′) bonded to the Sn(1′) related by symmetry, as well as a distance of 3.56(1) Å to the adjacent Cl(3) of Sn(2). The atom Sn(2) displays a distance of 3.81(1) Å to the adjacent Cl(1) atom bound to Sn(1), while Cl(4), bonded to the Sn(2), has no tin neighbors closer than 4 Å. The van der Waals radius sum [14] is 3.85 Å. The overall tin geometry might thus be construed to be an incipient linear tetramer of tin atoms Sn(1)–Sn(1′), 4.585(3) Å, Sn(1)–Sn(2), 4.726(4) Å incipiently doubly bridged in series by pairs of chloride atoms which display a nonsymmetric bridging geometry [incipient bond, 3.42(1)–3.80(1) Å; short bond 2.33(1)–2.39(1) Å]. The atom Sn(1) has pseudo six-coordinated geometry in this view, whereas Sn(2) is five-coordinated.

This structure, then, represents a tetrameric grouping which can be regarded as an incipient four-atom fragment of the one-dimensional associated polymer formed by dimethyl- [9] and diethyltin(IV) [10] dichloride.

We note that, except for those in bis(ferrocenyl)tin(IV) dichloride [4], the

Sn(1)–C_{Ph} distance of 2.05(3) Å is the shortest of those thus far determined for the diorganotin(IV) dichlorides [3–10]; while the Sn(2)–C_{Me} 2.29(4) Å is the longest.

The question of the veracity of the long Sn–Cl interactions may be addressed by considering the geometry of the two tin atoms. If the arrangement of the two Sn–Cl(close) and Sn–C bonds at each tin is roughly that of tetrahedral R_2SnCl_2, and if the arrangement is similar at each tin, then the long Sn–Cl distances are not indicative of significant bonding. If distortions are observed, the angles should be different at the tin atom assigned as five-coordinated Sn from those at the one assigned as six-coordinated.

The structure determination shows that both tin atoms display Cl(close)–Sn–Cl(close) angles of 98.6° and C–Sn–C angles that are similar (130(2) and 133(1)°). Variation in the Cl(close)–Sn–C angles is also similar at the two tin atoms 103.1(9)–109.0(8)° at Sn(1), 102(1)–108(1)° at Sn(2). This is precisely the variation expected on the basis of the principles of isovalent hybridization [32]. The long Sn–Cl interactions must not play a significant role in the coordination of the tin atoms in this structure because the geometries at the two tin atoms are identical within experimental limits. Moreover, as we learned from the monomeric structure of bis(biphenylyl-2)tin(IV) dichloride [3], even large distortions from tetrahedral geometry can be brought about by ligand steric demand combined with the dictates of isovalent rehybridization [32]. Distortion from tetrahedral angles at Sn may be viewed as being derived from the bulk of the phenyl group which opens the C–Sn–C angle and compresses the Cl–Sn–Cl angle. Hence, these arguments cannot be used to confirm or deny association by bridging atoms in the absence of other compelling evidence. We conclude that the tin atoms in the title compound are four coordinated. The organic groups lie on planes roughly perpendicular to the line joining the four tin atoms.

Table 4 lists comparison data for the seven other diorganotin(IV) dichlorides studied by X-ray diffraction [3–10] plus a modern electron-diffraction result for dimethyltin(IV) dichloride [33].

Tin-119m Mössbauer quadrupole splitting (QS) values generally increase with carbon–tin–carbon angles in diorganotin(IV) systems [34]. A treatment based upon a point charge model yields a quantitative estimate of the magnitude of this angle in dimethyl [35] and diphenyltin(IV) [36] derivatives using partial QS values assigned for the methyl and phenyl groups. Results are generally in accord where tests against known structural data can be made [1,2,37], but exceptions are known [38,39]. The model assumes that there will be no QS sign inversion in the systems being compared, and that the partial QS values for the ligands will be negligible compared to those of the organic groups whose magnitudes must remain constant over the range of compounds studied. Some recorded QS values exceed those predicting linear C–Sn–C angles [40,41] indicating breakdown of the model. Values greater than 6 mm s^{-1} have been recorded for Me_2Sn^{IV} systems [42]. Using an arithmetic average of the partial QS values assigned to methyl and phenyl yields and angle 135.3° for the carbon-tin-carbon angle (vs. 130(2) and 133(1)° measured), an error of only 3.3° from the mean or only 2.5%.

Experimental

Suitable crystals of the title compound, m.p. 44–45°C (lit. m.p. 43°C [16,19,20,23], 41–43°C [24], 46–47.5°C [25,30], 45.5°C [27]) subl. 35°C/0.01 torr,

TABLE 5

INFRARED AND RAMAN ABSORPTIONS (cm^{-1}) IN METHYLPHENYLTIN(IV) DICHLORIDE, $CH_3(C_6H_5)SnCl_2$ [a]

Infrared [b]	Raman
2930s	3050w
3860s	2925vw
1460s	1580m
1380m	1020w
770w	990s
730m	665m
690w	550s
540w	545s
440w	335s
350w	195m
250s	

[a] s = strong, m = medium, w = weak, v = very. [b] Assigned as 545s, $\nu(Sn-CH_3)$; 340m, 320m, $\nu(Sn-Cl)$; 255s, $\nu(Sn-C_6H_5)$ cm^{-1} in refs. 17, 20 and 23.

43–44 °C [28], 44.5 °C [17]), a known material from the HCl cleavage of methyltriphenyltin(IV) [20,23] were grown from the slow evaporation of a benzene solution.

Spectroscopic data include tin-119 Mössbauer (77 K), isomer shift (IS) = 1.52 ± 0.03; quadrupole splitting (QS) = 3.14 ± 0.06; $\Gamma_1 = 1.01 \pm 0.03$; $\Gamma_2 = 1.01 \pm 0.03$; ρ (= QS/IS) = 2.07 mm s^{-1} (lit. IS = 1.35; QS = 3.25 [23]; 1.44, 3.11 [26,27] mm s^{-1}) (recorded on a Ranger Scientific spectrometer equipped with a proportional counter and using $Ca^{119m}SnO_3$ as source and standard reference material for zero velocity. Velocity calibration was based upon β-tin and natural iron foils). NMR: $|^2J(^{119}Sn-C-^1H)| = 71.0$ ($CDCl_3$), 71.2 (C_6D_6), 87.9 [$(D_3C)_2CO$], 95.0 (CD_3OD) Hz; ^1H, δ 0.52 (C_6D_6) (3H), 7.1–7.8 (5H) ppm; (lit. δ 1.35 ppm, $|^2J|$ 72.1 Hz [18]; δ 7.5 m (5H), 1.23 s (3H) ppm, $|^2J|$ 68 Hz [25]; τ 8.62 ppm, 70.5 Hz [27]; δ 1.25 ppm, $|^2J|$ 71 Hz [28]; δ 1.25 ppm [30]) (vs. Me_4Si on an IBM NR-80 instrument). Mass spectrum (12 eV): *m/e* (fragment, assignment, relative abundance) 282 $\{CH_3C_6H_5SnCl_2]^{\ddagger}$ 33; 267 $[C_6H_5SnCl_2]^+$ 100; 247 $(CH_3C_6H_5SnCl]^{+'}$ 6; 205 $[CH_3SnCl_2]^+$ 3; 155 $[SnCl]^+$ 18; 135 $[CH_3Sn]^+$ 0.2; 120 $[Sn]^{\ddagger}$ 0.1 (lit. [30] (70 eV) 309 $[(C_6H_5)_2SnCl]^{2+}$ 2.4; 282 $[(CH_3)C_6H_5SnCl_2]^{\ddagger}$ 6.5; 267 $[C_6H_5SnCl_2]^+$ 41.6; 247 $[CH_3(C_6H_5)SnCl]^+$ 3.9; 232 $[C_6H_5SnCl]^{\ddagger}$ 0.5; 205 $[CH_3SnCl_2]^+$ 1.5; 197 $[C_6H_5Sn]^+$ 1.7; 155 $[SnCl]^+$ 35.6; 145 $[C_2HSn]^+$ 1.4; 120 $[Sn]^{\ddagger}$ 4.9) (Hewlett–Packard 5985B spectrometer). Mass numbers are based upon ^1H, ^{12}C, ^{35}Cl and ^{120}Sn. Infrared and Raman data are listed in Table 5.

Data collection and structure determination

A crystal of $CH_3(C_6H_5)SnCl_2$ was sealed in a capillary and mounted on a Syntex P3 automated diffractometer. Unit cell dimensions (Table 1) were determined by least-squares refinement of the best angular positions for fifteen independent reflections ($2\theta > 15°$) during normal alignment procedures using molybdenum radiation (λ 0.71069 Å). Data (6952 reflections) were collected at room temperature using a variable scan rate, a $\theta - 2\theta$ scan mode, and a scan width of 1.2° below K_{α_1} and 1.2° above K_{α_2} to a maximum 2θ value of 60.0°. Backgrounds were measured at each side of the scan for a combined time equal to the total scan time. The intensities of three standard reflections were remeasured after every 97 reflections and, as the intensities of these reflections showed less than 6% variation, corrections for decomposition were deemed unnecessary. Data were corrected for Lorentz, polarization and background effects. After removal of redundant and space-group forbidden data, 1793 reflections were considered observed ($I > 3.0\sigma(I)$). The structure was solved using MULTAN80 [43]. Refinement of scale-factor [44], positional and anisotropic thermal parameters for all non-hydrogen atoms was carried out to convergence. Hydrogen-atom positions were not determined. The final cycle of refinement (function minimized: $\Sigma(|F_o| - |F_c|)^2$) led to a final agreement factor, $R = (\Sigma || F_o| - |F_c|| / \Sigma |F_o|)$ 0.085. Anomalous dispersion corrections were made for Sn and Cl. Scattering factors were taken from ref. 45. Unit weights were used until the final cycles of the refinement when a weight of $1/\sigma F$ was introduced; $R_w = 0.131$.

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Supplementary Material Available. Anisotropic thermal parameters, complete bond angles and distances and structure factors (51 pages) is available from the authors.

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