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THE CRYSTAL AND MOLECULAR STRUCTURE OF TRIS[(TRIPHENYLSTANNYL)METHYL]METHANE

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Summary

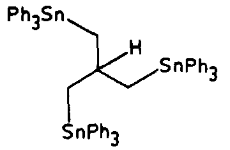
The title compound, $C_{58}H_{52}Sn_3$, belongs to the triclinic space group $P\bar{1}$, with *a* 10.165, *b* 13.365, *c* 18.670 Å, α 96.28, β 93.88, γ 103.15°, $V = 2443.8$ Å^3, $f_w = 1105.1$, $Z = 2$, D_{calc} 1.501 g cm^{-3}, m.p. 206.5–208°C, λ(Mo-$K_{\bar{\alpha}}$) 0.71069 Å. The structure was refined on 2684 nonzero reflections to an *R* factor of 0.044. The crystal contains molecules in which the $(SnCH_2)_3CH$ core possesses an approximate C_3 symmetry. The three Sn–C(H_2) bonds are *gauche* to the C(4)–H bond. Repulsive interactions involving the bulky Ph_3Sn substituents lead to large Sn–C(H_2)–C(H) angles (av. 117.3°), whereas the C(H_2)–C(H)–C(H_2) angles at the tertiary carbon average 111.3°. Little distortion of the Ph_3Sn groups themselves is present, since the Ph–Sn–Ph angles (av. 109.8°) are almost equal to the C(H_2)–Sn–Ph angles (av. 109.9°). The molecule as a whole has no symmetry because the aromatic rings in the three Ph_3Sn groups have different orientations. The phenyl groups create a pocket in the middle of the molecule which encloses and shields the tertiary hydrogen atom. The resulting inaccessibility of this hydrogen accounts in part for the low reactivity of the title compound in redox reactions.

Introduction

Compounds in which a carbon–hydrogen bond is adjacent to several carbon–metal bonds promise to be good reducing agents, since transfer of hydrogen may produce a carbocation or a radical highly stabilized by hyperconjugation (eq. 1) [1,2].

(1)

Stannane **1** [3] only partially fulfills this promise, since its reduction of triphenylcarbenium salts to triphenylmethane is unexpectedly slow [3]. Molecular models strongly suggest that the large triphenyltin groups [4] of stannane **1** will force the molecule to adopt the enantiomeric C_3 conformations *2* and *2'*. This preference



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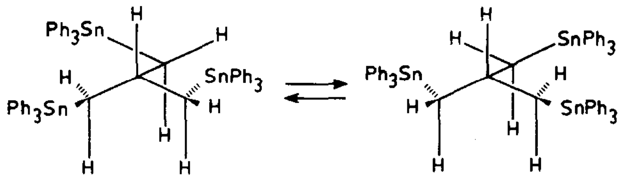

2 2'

would have two important and unfortunate consequences. One is that the electronically activated methine hydrogen would be shielded by three triphenyltin groups and made sterically inaccessible. The second is that all of the carbon–tin bonds would be *gauche* to the central carbon–hydrogen bond, whereas loss of hydrogen should be fastest when the carbon–metal and carbon–hydrogen bonds are *anti*-periplanar [5]. To confirm our suggestion [3] that the low reactivity of stannane **1** is due to a preference for conformations **2** and **2'** in solution, we decided to study the structure of compound **1** in the solid state.

Experimental

Crystal data: $C_{58}H_{52}Sn_3$; fw = 1105.1; triclinic, space group $P\bar{1}$, a 10.165(3), b 13.365(9), c 18.670(18), Å, α 96.28(7), β 93.88(5), γ 103.15(4)°, V 2443.8 Å^3, D_{calc} 1.501 g cm^{-3}, $Z = 2$, λ(Mo-$K_{\bar{\alpha}}$) 0.71069 Å (graphite monochromator), μ(Mo-K_α) 15.6 cm^{-1}, T 23°C.

Crystallographic measurements

The compound was prepared from 3-chloro-2-chloromethyl-1-propene by a five-step synthesis, as described earlier (m.p. 206.5–208°C) [3]. Suitable colorless crystals were grown at 25°C from a 1/1 mixture of hexane and ethyl acetate. The specimen used for X-ray work had the following dimensions between the indicated pairs of faces: 0.04 mm (001-00$\bar{1}$) × 0.12 mm (100-$\bar{1}$00) × 0.24 mm (011-0$\bar{1}\bar{1}$). The crystal was mounted on an Enraf–Nonius CAD4 diffractometer with the longest direction along the ϕ axis. A list of 25 reflections randomly distributed in the diffraction sphere was created by the automatic search procedure of the CAD4 software. These reflections were centered and the autoindexing procedure yielded the triclinic reduced cell given above. Long-exposure oscillation photographs were recorded about each of the crystallographic axes, to make sure that no intermediate levels had been missed. Inspection of the Niggli parameters [6] clearly ruled out the possibility of describing the crystals with higher symmetry lattice.

The intensity data were collected as described elsewhere [7]. A total of 6362 independent reflections (hkl, $hk\bar{l}$, $h\bar{k}l$ and $h\bar{k}\bar{l}$, $2\theta \leqslant 45°$) were collected by using graphite-monochromated Mo-$K_{\bar{\alpha}}$ radiation. The fluctuation on three standard reflections remained within ±3% during the experiment. A set of 2684 reflections significantly above background ($I > 2.5\ \sigma(I)$) was retained for structure determination, and these data were corrected for the Lorentz and polarization effects. Calculation of the absorption correction on randomly distributed sample reflections indicated that the transmission coefficient would lie in the narrow 0.84–0.91 range, so absorption correction was deemed unnecessary.

Resolution of the structure

The structure was solved by the heavy-atom method and refined by full-matrix least squares in the early stages. A three-dimensional Patterson map revealed the positions of the three independent Sn atoms. The remaining nonhydrogen atoms were located from a series of structure factor and difference Fourier (ΔF) map calculations. Isotropic refinement using unit weights led to the discrepancy factor $R = \Sigma||F_o| - |F_c||/\Sigma|F_o| = 0.075$. Anisotropic temperature factors were then assigned to the nonhydrogen atoms. The hydrogen atoms were positioned at the ideal coordinates, with isotropic B 6.0 Å^2. They were not refined, but their coordinates were recalculated after each least-squares cycle. In the last cycles, individual weights based on counting statistics were applied to each reflection and refinement was carried out by block-diagonal least squares. Anisotropic refinement of all nonhydro-

TABLE 1

REFINED COORDINATES ($\times 10^3$, Sn $\times 10^4$)[a]

Atom	x	y	z	Atom	x	y	z
Sn(1)	3312(1)	691(1)	2341(1)	C(51)	11(2)	−329(1)	130(1)
Sn(2)	2004(1)	−3097(1)	834(1)	C(52)	−6(2)	−360(1)	196(1)
Sn(3)	3817(1)	−2803(1)	3615(1)	C(53)	−128(2)	−373(2)	228(1)
C(1)	423(2)	−56(1)	258(1)	C(54)	−240(2)	−356(2)	188(1)
C(2)	343(2)	−175(1)	142(1)	C(55)	−225(2)	−329(1)	124(1)
C(3)	415(2)	−246(1)	253(1)	C(56)	−106(2)	−311(1)	93(1)
C(4)	350(2)	−164(1)	224(1)	C(61)	265(2)	−449(1)	89(1)
C(11)	319(2)	84(1)	122(1)	C(62)	398(2)	−455(1)	84(1)
C(12)	202(2)	80(1)	83(1)	C(63)	436(2)	−547(2)	87(1)
C(13)	192(2)	92(1)	10(1)	C(64)	346(2)	−635(1)	96(1)
C(14)	310(2)	105(1)	−25(1)	C(65)	215(2)	−633(1)	102(1)
C(15)	431(2)	111(2)	14(1)	C(66)	177(2)	−540(1)	101(1)
C(16)	431(2)	102(1)	84(1)	C(71)	565(2)	−301(1)	416(1)
C(21)	125(2)	48(1)	266(1)	C(72)	566(2)	−343(2)	476(1)
C(22)	88(2)	118(1)	313(1)	C(73)	687(2)	−355(2)	515(1)
C(23)	−44(2)	103(2)	330(1)	C(74)	804(2)	−319(2)	486(1)
C(24)	−142(2)	16(2)	297(1)	C(75)	808(2)	−279(2)	428(1)
C(25)	−105(2)	−53(2)	251(1)	C(76)	689(2)	−264(2)	390(1)
C(26)	28(2)	−37(1)	232(1)	C(81)	304(2)	−163(1)	423(1)
C(31)	461(2)	206(1)	293(1)	C(82)	366(2)	−110(1)	489(1)
C(32)	447(2)	302(2)	276(1)	C(83)	311(2)	−36(2)	523(1)
C(33)	535(2)	394(1)	311(1)	C(84)	198(2)	−16(2)	496(1)
C(34)	632(2)	387(2)	362(1)	C(85)	131(2)	−65(2)	433(1)
C(35)	649(2)	293(2)	374(1)	C(86)	184(2)	−140(1)	396(1)
C(36)	563(2)	206(1)	341(1)	C(91)	228(2)	−417(1)	357(1)
C(41)	185(2)	−279(1)	−27(1)	C(92)	126(2)	−427(1)	402(1)
C(42)	168(2)	−187(1)	−46(1)	C(93)	19(2)	−512(2)	396(1)
C(43)	167(2)	−166(2)	−117(1)	C(94)	10(2)	−596(2)	342(1)
C(44)	192(2)	−237(2)	−170(1)	C(95)	109(2)	−589(1)	297(1)
C(45)	205(2)	−330(2)	−155(1)	C(96)	218(2)	−503(1)	304(1)
C(46)	203(2)	−349(1)	−85(1)				

[a] Standard deviation on the last digit given within parentheses in this and the following tables.

gen atoms converged to $R = 0.044$ and $R_w = [\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2]^{1/2} = 0.048$. The goodness-of-fit ratio was 1.39. The final ΔF map was essentially featureless, the highest residuals (± 0.4 e/Å^3) being found near Sn atoms.

The refined coordinates are listed in Table 1. Lists of temperature factors, ideal positions of the phenyl hydrogens, and structure factor amplitudes have been deposited *.

The scattering curves used were those of Cromer and Waber [8], except for hydrogen [9]. The f' and f'' contributions to anomalous dispersion of Sn were taken into account [8]. The programs used are listed elsewhere [10].

Description of the structure and discussion

A stereoview of the molecule down the tertiary C(4)–H bond is shown in Fig. 1. Selected interatomic distances and bond angles are listed in Table 2.

Magnetic equivalence of the three tin atoms and the hydrogen atoms of the three methylene groups in solution at 25°C was deduced from the ^{119}Sn and ^{1}H NMR spectra and suggested an instantaneous or time-averaged three-fold axis of symmetry [3]. In the solid state, no particular symmetry is imposed by the space group. The molecule is devoid of a C_3 axis, mainly because the orientations of the phenyl rings differ in the three Ph_3Sn groups (Fig. 1). However, the $(SnCH_2)_3CH$ core does possess an approximate three-fold axis running through the C(4)–H bond. Indeed, corresponding bond lengths and bond angles are equal within experimental errors (Table 2). Furthermore, the C(H$_2$)–C(H)–C(H$_2$)–Sn torsion angles (Table 3) correspond to similar conformations in the three chains. There are significant differences between these three angles, which should ideally be equal, but the differences lie

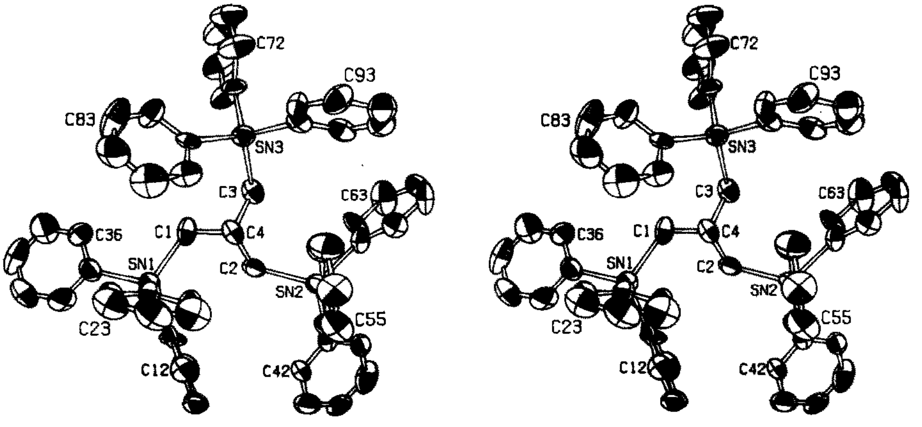

Fig. 1. Stereoview of the $(Ph_3SnCH_2)_3CH$ molecule down the approximate 3-fold axis (C(4)–H(1) bond). Hydrogens are omitted for simplicity. The ellipsoids correspond to 50% probability.

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TABLE 2

SELECTED INTERATOMIC DISTANCES AND BOND ANGLES

Distances (Å)			
Sn(1)–C(1)	2.164(17)	Sn(3)–C(3)	2.157(16)
Sn(1)–C(11)	2.122(17)	Sn(3)–C(71)	2.150(18)
Sn(1)–C(21)	2.183(18)	Sn(3)–C(81)	2.166(15)
Sn(1)–C(31)	2.136(18)	Sn(3)–C(91)	2.106(17)
Sn(2)–C(2)	2.169(17)	C(1)–C(4)	1.51(2)
Sn(2)–C(41)	2.148(17)	C(2)–C(4)	1.52(2)
Sn(2)–C(51)	2.140(18)	C(3)–C(4)	1.53(2)
Sn(2)–C(61)	2.121(16)	C–C (phenyl)	1.37(3) [a]

Angles (deg)			
C(1)–Sn(1)–C(11)	112.1(6)	Sn(3)–C(3)–C(4)	118.0(11)
C(1)–Sn(1)–C(21)	112.5(6)	C(1)–C(4)–C(2)	111.9(14)
C(1)–Sn(1)–C(31)	105.3(7)	C(1)–C(4)–C(3)	111.4(14)
C(11)–Sn(1)–C(21)	106.6(6)	C(2)–C(4)–C(3)	110.5(14)
C(11)–Sn(1)–C(31)	109.6(7)	C(3)–Sn(3)–C(71)	110.3(6)
C(21)–Sn(1)–C(31)	110.7(7)	C(3)–Sn(3)–C(81)	112.1(6)
C(2)–Sn(2)–C(41)	105.5(6)	C(3)–Sn(3)–C(91)	109.4(7)
C(2)–Sn(2)–C(51)	109.0(7)	C(71)–Sn(3)–C(81)	111.1(6)
C(2)–Sn(2)–C(61)	113.0(6)	C(71)–Sn(3)–C(91)	109.3(7)
C(41)–Sn(2)–C(51)	112.8(6)	C(81)–Sn(3)–C(91)	104.4(6)
C(41)–Sn(2)–C(61)	109.8(6)	C_o–C_{Sn}–C_o	116.2(15) [a]
C(51)–Sn(2)–C(61)	106.8(7)	C_{Sn}–C_o–C_m	122.0(15) [a]
Sn(1)–C(1)–C(4)	116.9(12)	C_o–C_m–C_p	119.9(15) [a]
Sn(2)–C(2)–C(4)	116.9(11)	C_m–C_p–C_m	120.0(15) [a]

[a] average values

within a range that can reasonably be ascribed to crystal packing.

The three Sn–C(H$_2$) bonds are *gauche* to the C(4)–H bond, the Sn–C(H$_2$)–C(H)–H angles averaging −49°C. This arrangement appears to mini-

TABLE 3

SELECTED TORSION ANGLES (deg) [a]

C(1)–C(4)–C(2)–Sn(2)	−165(1)	H(1)–C(4)–C(1)–Sn(1)	−54 [b]
C(2)–C(4)–C(3)–Sn(3)	−161(1)	H(1)–C(4)–C(2)–Sn(2)	−49 [b]
C(3)–C(4)–C(1)–Sn(1)	−173(1)	H(1)–C(4)–C(3)–Sn(3)	−43 [b]
C(1)–C(4)–C(3)–Sn(3)	74(2)	C(1)–Sn(1)–C(11)–C(16)	−59(2) [c]
C(2)–C(4)–C(1)–Sn(1)	63(2)	C(1)–Sn(1)–C(21)–C(26)	−63(2)
C(3)–C(4)–C(2)–Sn(2)	70(2)	C(1)–Sn(1)–C(31)–C(36)	−9(2)
C(4)–C(1)–Sn(1)–C(11)	−62(1)	C(2)–Sn(2)–C(41)–C(42)	−47(2)
C(4)–C(1)–Sn(1)–C(21)	58(1)	C(2)–Sn(2)–C(51)–C(52)	−71(2)
C(4)–C(1)–Sn(1)–C(31)	179(1)	C(2)–Sn(2)–C(61)–C(62)	−41(2)
C(4)–C(2)–Sn(2)–C(61)	−73(1)	C(3)–Sn(3)–C(71)–C(76)	18(2)
C(4)–C(2)–Sn(2)–C(51)	45(1)	C(3)–Sn(3)–C(81)–C(86)	57(2)
C(4)–C(2)–Sn(2)–C(41)	167(1)	C(3)–Sn(3)–C(91)–C(96)	39(2)
C(4)–C(3)–Sn(3)–C(71)	−140(1)		
C(4)–C(3)–Sn(3)–C(81)	−15(1)		
C(4)–C(3)–Sn(3)–C(91)	100(1)		

[a] Torsion angles defined according to ref. 46. [b] Ideal position for H(1) used. [c] Ring orientation defined with the ortho carbon atom giving the torsion angle closer to 0°.

TABLE 4

COMPARISONS OF Sn–alkyl AND Sn–aryl BOND LENGTHS [a]

Molecules of the same stoichiometry

Formula	Phase	Bond length Sn–C(Å)	Formula	Phase	Bond length Sn–C(Å)	Ref.
$(CH_3)_4Sn$	Gas	2.144(3)	Ph_4Sn	Solid	2.144(14)	13,14
			$(p\text{-Tolyl})_4Sn$	Solid	2.147(6)	15
$(CH_3)_3SnCl$	Gas	2.106(6)	Ph_3SnCl	Solid	2.122(15)	16,17
$(CH_3)_2SnCl_2$	Gas	2.108(7)	Ph_2SnCl_2	Solid	2.113(6)	18,19
$[(CH_3)_3Sn]_2O$	Gas	2.170(5)	$[Ph_3Sn]_2O$	Solid	2.135(3)	20,21
$(CH_3)_3SnMn(CO)_5$	Solid	2.130(15)	$Ph_3SnMn(CO)_5$	Solid	2.15(4)	22,23

Solids with aryl and alkyl groups in the same molecule

Formula	Bond lengths		Ref.
	Sn–C(alkyl)	Sn–C(aryl)	
$Ph_3SnCH(CH_3)(C_2H_5)$	2.22(2)	2.15(1)	24
Ph_3SnCH_2I	2.134(6)	2.133(6)	25
$PhSn(CH_3)_2C(Ph)=C(Ph)C(Ph)=C(Ph)Cl$	2.167(11)	2.116(9)	27
$PhSn(CH_3)_2C(Ph)=C(Ph)C(Ph)=C(Ph)Br$	2.176(14)	2.141(13)	27
$Ph_2SnC_4H_8Sn(Ph)_2C_4H_8$	2.161(8)	2.141(8)	28
$(Ph_3SnCH_2)_3CH$	2.157(16)	2.137(16)	This work

[a] Average values (average esd).

mize the interactions of the Ph_3Sn groups, but there is still considerable crowding. The main result of this crowding is enlargement of the C(H)–C(H_2)–Sn angles, which are, on the average, 7.8° greater than the normal tetrahedral value. The fact that the central C(H_2)–C(H)–C(H_2) angles (av. 111.3°) are slightly larger than 109.5° probably has a similar origin, since the small size of hydrogen allows the three substituents at C(4) to move away from one another [11].

As expected, the environment of tin is tetrahedral [12]. The Sn–C(H_2) distances (av. 2.163(17) Å) seem to be slightly longer than the Sn–Ph distances (av. 2.141(16) Å). The results available in the literature for pairs of corresponding alkyl and aryl compounds were generally obtained in the gas phase for the alkyl compound and in the solid state for the aryl derivative (Table 4). This makes direct comparisons hazardous. A few structures are known in which both alkyl and aryl groups are present within the same molecule. In these compounds, the Sn–aryl bonds tend to be 0.02–0.03 Å shorter than the Sn–alkyl bonds, and this is the trend observed here as well.

It was also of interest to estimate the effect of crowding on the $SnPh_3$ groups by comparing the angles around tin with those observed in other compounds. The data in Table 5 show that in Ph_3SnX molecules with small X groups, repulsion among the phenyl rings increases the Ph–Sn–Ph angles significantly above 109.5°, with an upper limit of ~ 114°. When the group X is a hydrocarbon residue, the Ph–Sn–Ph angle is a function of the bulk of this residue: for example, the Ph–Sn–Ph angle averages 109.5° in Ph_4Sn, decreases to 107.3° in a hindered naphthyl derivative, but reaches 111.0° when X is a straight diyne chain which occupies less space. For Ph_3Sn groups bonded to a transition metal, the high coordination number of the

TABLE 5

AVERAGE ANGLES AROUND TIN IN Ph_3SnX COMPOUNDS

Angle Ph–Sn–X	Angle Ph–Sn–Ph	X	Ref.
105.2(2)	113.4(3)	Br	29
105.6(5)	113.4(6)	Cl	17
106.0(1)	112.7(1)	Ph_3SnO	21
103.7(5)	114.6(6)	$(CO)_3Mn(Ph_4C_5O)$ [a]	30
108.2(3)	110.7(4)	4-(t-Bu)C_6H_4S	31
107.9(8)	110.9(10)	*o*-$CH_3C_6H_4S$	32
106.8	112.0	6-[1,5-Br_2-3-FC_6H_2S]	32
107.8(1)	111.1(1)	$SP(OEt)_2S$	33
106.1(5)	112.6(6)	2-[1,3,5-$(CH_3)_3$-C_6H_2S]	34
109.6(4)	109.5(8)	Ph_3SnSe	35
110.3(5)	108.7(6)	Ph_3Sn	44
118(2)	100(3)	$[(Ph_2PC_2H_4)_3N]Ni$	36
115.9(4)	102.3(5)	$L(CO)_4Cr$ [b]	37
114(1)	105(2)	$(Ph_3P)(CO)_4Mn$	38
106.0(19)	112.7(13)	$(CO)_5Mn$	39
112.8(2)	105.8(3)	$(Ph_3Sn)(CO)_4Fe$	40
115.7(5)	102.6(6)	$(Ph_3Sn)(CO)_5V$	41
116.4(5)	101.7(7)	$(Ph_3Sn)_2(NO_3)Sn$	45
118.3(4)	102.0(5)	$(NO_3)Sn$	45
109.5(7)	109.5(7)	Ph	14
109.3(3)	109.6(2)	ICH_2	25
112.6(14)	106.1(10)	7-cyclohepta-1,3,5-trienyl	42
107.9(3)	111.0(3)	$Ph_3SnC{\equiv}CC{\equiv}C$	26
111.5(4)	107.3(4)	3-[4-(N-methylanilino)-1-Ph-2-naphthol]	43
111.1(1)	107.8(1)	$C_2H_5CH(CH_3)$	24

[a] Triphenylstannoxytetraphenylcyclopentadienyltricarbonylmanganese. Sn bonded to oxygen on the 5-membered carbon ring. [b] L = diethylaminocarbyne.

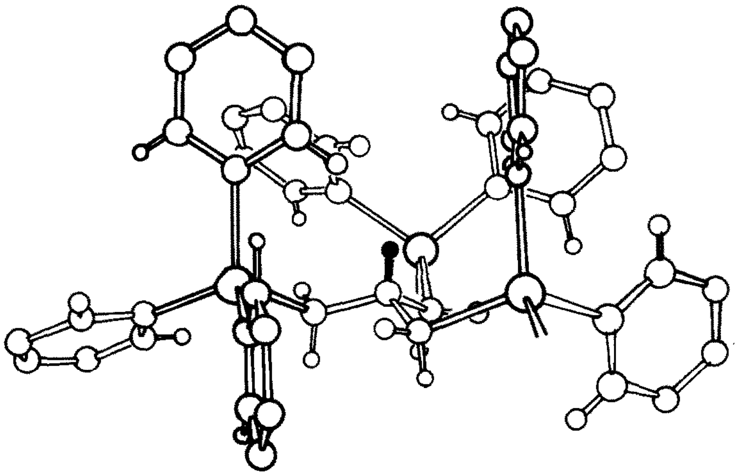

Fig. 2. Pocket containing the C(4)–H(1) bond. Only the *ortho*-hydrogens are shown for the phenyl rings.

metal often produces angles as small as 100°, although in compounds like $Mn(CO)_5(SnPh_3)$, where the carbonyl ligands are possibly more easily pushed away from the Ph_3Sn moiety, a large angle of 112.7° is found. We find in stannane **1** that the angles Ph–Sn–Ph average 109.8°, so steric factors lead to little distortion of the Ph_3Sn groups.

The C(H)–$C(H_2)$–Sn–C torsion angles (Table 3) show that two of the three Ph_3Sn groups (those containing Sn(1) and Sn(2)), adopt similar arrangements of the Sn–Ph bonds with respect to the central core. However, the orientations of the individual rings, as measured by the $C(H_2)$–Sn–C–C(*ortho*) angles nearer 0°, do not follow a definite pattern. These torsion angles are undoubtedly among the structural parameters which are easiest to change, and thus they are highly sensitive to packing forces. The geometry of the phenyl rings is normal (Table 2). The rings are all planar within 1.5 σ (0.03 Å), but the Sn atom often deviates considerably from this plane (maximum Sn-to-plane distance 0.18 Å).

The overall arrangement of the Ph_3Sn groups in stannane **1** creates a pocket in the middle of the molecule which encloses the tertiary hydrogen atom. This hydrogen is shielded from its surroundings by four of the nine phenyl rings (Fig. 2). The resulting inaccessibility of this hydrogen accounts in part for the low reactivity of stannane **1** in redox reactions [3].

Acknowledgment

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