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C-Stannylated carbohydrate derivatives

IV. * Structure and reactivity of methyl 4,6-*O*-benzylidene-3-deoxy-3-triphenylstannyl-α-D-altropyranoside

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Abstract

The crystal structure of orthohombic methyl 4,6-*O*-benzylidene-3-deoxy-3-triphenylstannyl-α-D-altropyranoside (II) has been determined (space group $P2_12_12_1$). In the solid state II adopts a $^4C_1$ conformation. The bond angles about tin are close to those expected for a tetrahedral arrangement, however there are two intramolecular Sn- - -O contacts, with O(1) and O(4) at distances of 3.25(2) and 3.23(2) Å respectively.

Compound II is decomposed by strong Bronsted acids, e.g. CF_3CO_2H, but reacts straightforwardly with halogens with phenyl–tin bond cleavage. Nucleophilic assistance provided by both or either of O(1) or O(4) would account for the enhanced reactivity of II towards iodine.

Introduction

A particular and intriguing group of functionally-substituted organotin compounds is formed by the tin–carbon bonded carbohydrate derivatives. Studies of these compounds have concentrated mainly on their syntheses and reactivities [1–10], with little investigation being made on structures [4,6]. The single crystal structure of only one such compound has sofar been published [6]; this was of 3-*C*-(dibutyliodostannyl)methyl-1,2 : 5,6-di-*O*-isopropylidene-α-D-allofuranose (I).

* Part III see ref. 4.

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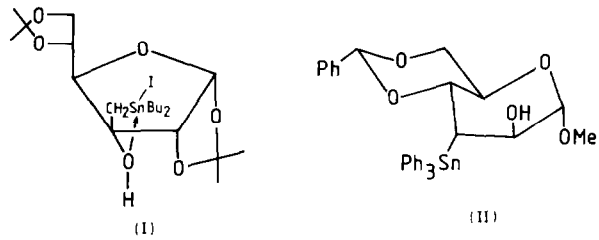


Fig. 1. Suggested conformation of II in C_6D_6 solution.

Conformations of other stannyl monosaccharides in C_6D_6 solution have been deduced from NMR coupling constant data [6]; for example a $^{\circ}S_5$ conformation was assigned to methyl 4,6-*O*-benzylidene-3-deoxy-3-triphenylstannyl-α-D-altropyranoside (II) (Fig. 1).

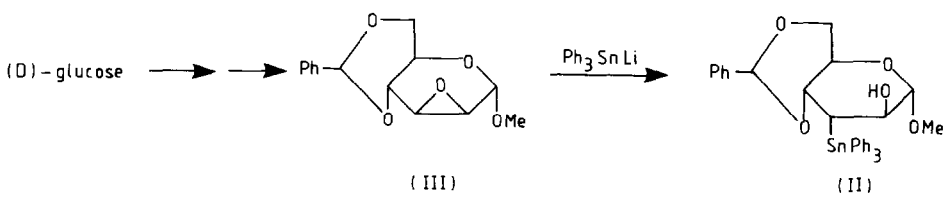
In order to determine whether this conformation pertains in the solid state, the crystal and molecular structures of II has been investigated. In addition to the structural data, we also wish to report on the reactivity of II towards some electrophilic reagents.

Results and discussion

Compound II was obtained from (D)-glucose, by a modification of the procedure used by Hall et al. [6] (Scheme 1). The opening of the epoxide ring in III by Ph_3SnLi occurs regiospecifically to give II: no other isomer was obtained. Compound II is stable to air and moisture; the parent peak (1%) was observed in the mass spectrum at 20 eV.

Crystal structure of II. The single crystal data of II are consistent with the atomic arrangement shown in Fig. 2. Atomic coordinates are listed in Table 1, bond lengths in Table 2 and valency angles in Table 3. The conformation adopted by the ring system of II in the solid state is $^4C_1$ and is different from that deduced earlier for the solution conformation from NMR data (see later) [6].

The Sn–C bond lengths in II range from 2.149(15) to 2.191(18) Å and are in the expected region. There are two Sn- - -O intramolecular distances of 3.25(2) Å with the OMe [O(1)] and 3.23(2) Å with the acetal O [O(4)]. These distances are considerably larger than the sum of the covalent radii (2.11 Å) but are within the sum of the Van der Waal radii (3.70 Å). The normal Sn–O covalent bond is ca. 2.0 Å [11]. Intramolecular Sn- - -O distances in the range from 2.263(6) to 3.071(2) Å have been confidently reported to indicate Sn–O bonding [4,12–16]; indeed even for greater Sn- - -O distances, e.g. (3.206(3) Å in Ph_3SnOAc [11], it has been concluded



Scheme 1.

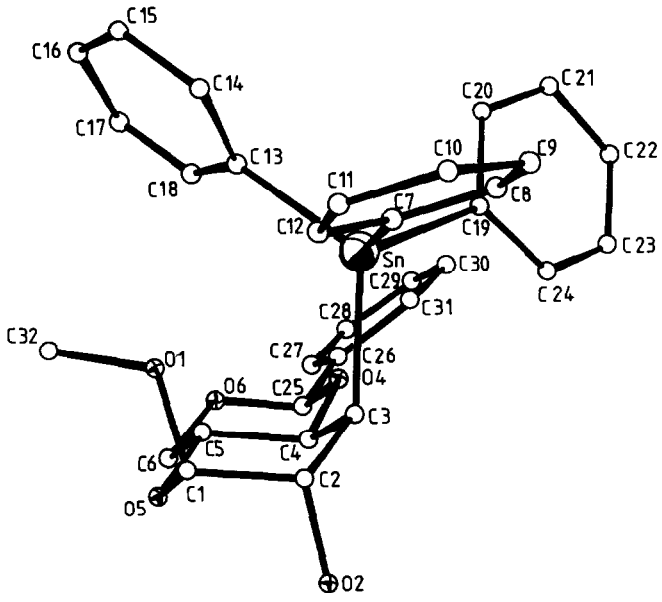
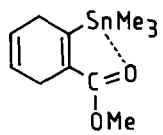


Fig. 2. Molecular structure and atom numbering scheme for compound II.

that Sn- - -O bonding occurs. The Sn - - -O intramolecular distance in the pentacoordinate tin-carbohydrate species (I) is 2.68(2) Å; the bond angles at tin in I clearly show a distortion away from those expected for a tetrahedral arrangement towards those required for a trigonal bipyramid geometry. In contrast, the bond angles at tin in II are only slightly distorted from a tetrahedral array, being in the range 105.2(7) to 108.9(5)°, except that for C(13)–Sn–C(3), which has the value of 120.6(6)° (Table 3). The angles formed by O(1)- - -Sn–C(19) and O(4)- - -Sn–C(7) are 164.2(5) and 155.8(5)° respectively; whether these angles arise to accommodate Sn–O coordination remains unresolved.

Normally tetraorganotin compounds contain tetracoordinate tin atoms; however a few functionally-substituted tetraorganotin compounds having a more highly coordinated tin atom are known, with either oxygen as the additional donor atom, e.g. in compound IV [16], or nitrogen as the donor atom, e.g. in {[3-(2-pyridyl)-2-thienyl]Sn(C_6H_4Me-*p*)$_3$} (5-coordinate Sn) [17], {[3-(2-pyridyl)-2-thienyl]$_2$SnPh$_2$} (6-coordinate Sn) [18] and the stannantrane, MeSn(CH_2CH_2)$_3$N (5-coordinate Sn) [19]. The intramolecular Sn- - -N distances (2.56–2.84 Å) in these compounds and the Sn- - -O distance, in IV, are considerably shorter than the Sn- - -O contacts found in II.



(IV) Sn----O 2.781 Å

Solution study. The δ ^{119}Sn value for II in $CDCl_3$ is −118.4 ppm and is as expected for a four coordinate tin centre in an alkyltriphenylstannane derivative, c.f. −118.9 ppm for Ph_3SnCH_2I.

Table 1

Fractional atomic coordinates and isotropic (or equivalent isotropic) temperature factors for compound (II) with e.s.d.s in parenthesis

	x	y	z	U_{eq}/U_{iso} [a]
Sn	−0.24310(19)	−0.09998(9)	−0.80599(6)	0.037(6)
O(1)	−0.3500(15)	−0.1503(13)	−0.9472(7)	0.068(11)
O(2)	−0.5220(14)	0.0739(10)	−0.9219(7)	0.061(11)
O(4)	−0.5560(11)	−0.1220(9)	−0.7615(5)	0.034(1)
O(5)	−0.5856(13)	−0.1298(10)	−0.9333(6)	0.051(9)
O(6)	−0.7074(13)	−0.2444(10)	−0.7877(6)	0.048(1)
C(1)	−0.458(2)	−0.086(2)	−0.950(1)	0.061(1)
C(2)	−0.4266(18)	0.0029(16)	−0.9086(9)	0.041(1)
C(3)	−0.4273(18)	−0.0256(13)	−0.8376(10)	0.032(5)
C(4)	−0.5575(19)	−0.0814(13)	−0.8243(8)	0.038(1)
C(5)	−0.582(2)	−0.163(2)	−0.869(1)	0.049(1)
C(6)	−0.715(2)	−0.216(2)	−0.853(1)	0.054(2)
C(7)	−0.0734(14)	−0.0316(10)	−0.8524(5)	0.043(7)
C(8)	0.0098(14)	0.0326(10)	−0.8190(5)	0.049(6)
C(9)	0.1192(14)	0.0775(10)	−0.8492(5)	0.081(8)
C(10)	0.1455(14)	0.0581(10)	−0.9126(5)	0.062(7)
C(11)	0.0623(14)	−0.0061(10)	−0.9459(5)	0.064(6)
C(12)	−0.0472(14)	−0.0510(10)	−0.9158(5)	0.047(6)
C(13)	−0.2200(11)	−0.2544(9)	−0.8190(6)	0.040(5)
C(14)	−0.0994(11)	−0.2865(9)	−0.8466(6)	0.054(6)
C(15)	−0.0802(11)	−0.3854(9)	−0.8588(6)	0.069(7)
C(16)	−0.1816(11)	−0.4522(9)	−0.8432(6)	0.057(7)
C(17)	−0.3023(11)	−0.4202(9)	−0.8156(6)	0.057(6)
C(18)	−0.3215(11)	−0.3212(9)	−0.8034(6)	0.044(5)
C(19)	−0.2306(14)	−0.0713(8)	−0.7064(7)	0.047(6)
C(20)	−0.1522(14)	−0.1329(8)	−0.6687(7)	0.083(9)
C(21)	−0.1494(14)	−0.1199(8)	−0.6035(7)	0.080(3)
C(22)	−0.2250(14)	−0.0453(8)	−0.5758(7)	0.070(7)
C(23)	−0.3035(14)	0.0164(8)	−0.6135(7)	0.065(7)
C(24)	−0.3063(14)	0.0033(8)	−0.6787(7)	0.053(6)
C(25)	−0.681(2)	−0.168(1)	−0.749(1)	0.042(1)
C(26)	−0.6816(19)	−0.2028(16)	−0.6817(8)	0.037(1)
C(27)	−0.589(2)	−0.173(1)	−0.637(1)	0.045(1)
C(28)	−0.602(3)	−0.195(2)	−0.573(1)	0.070(2)
C(29)	−0.715(3)	−0.248(2)	−0.556(1)	0.070(2)
C(30)	−0.809(2)	−0.280(2)	−0.599(1)	0.075(2)
C(31)	−0.789(3)	−0.255(2)	−0.660(1)	0.066(2)
C(32)	−0.369(3)	−0.237(2)	−0.983(1)	0.077(2)

[a] $U_{eq} = 1/3 \sum_i \sum_j a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$.

Hall et al. [6] deduced the $^{\circ}S_5$ skew conformation for II (Fig. 1) in C_6H_6 solution from ^1H and ^{13}C NMR data, in particular the values of specific coupling constants, e.g. $J(H_3-H_4)$ (Table 4). The skew conformation was assumed to arise because of interactions involving the bulky Ph_3Sn unit. Our ^1H NMR spectrum of II in C_6D_6 is in agreement with the reported by Hall et al. [6]; however we believe that they have wrongly assigned δ H_2 and δ OH and that the values they quote should be interchanged (i.e. to give δ H_2 4.16 and δ OH 1.42 ppm). The spectrum in $CDCl_3$ is basically the same as that in C_6D_6, apart from specific solvation shifts, particularly

Table 2

Bond lengths (Å) for compound II with e.s.d.s in parenthesis

Sn–C(3)	2.185(18)	Sn–C(7)	2.152(14)
Sn–C(13)	2.149(13)	Sn–C(19)	2.152(15)
O(1)–C(1)	1.38(3)	O(1)–C(32)	1.42(4)
O(2)–C(2)	1.38(3)	O(4)–C(4)	1.44(2)
O(4)–C(25)	1.40(3)	O(5)–C(1)	1.43(3)
O(5)–C(5)	1.43(3)	O(6)–C(6)	1.43(3)
O(6)–C(25)	1.36(3)	C(1)–C(2)	1.54(4)
C(2)–C(3)	1.55(3)	C(3)–C(4)	1.52(3)
C(4)–C(5)	1.50(3)	C(5)–C(6)	1.53(3)
C(7)–C(8)	1.395(19)	C(7)–C(12)	1.395(16)
C(8)–C(9)	1.395(19)	C(9)–C(10)	1.395(16)
C(10)–C(11)	1.395(19)	C(11)–C(12)	1.395(19)
Sn- - -O(1)	3.25(2)	Sn- - -O(4)	3.23(2)

for the OH, H_5, H_6 and $H_6{}^1$ protons (see Table 4). A D_2O shake of the solution of (II) in $CDCl_3$ resulted in a fairly slow exchange of OH to OD with the removal of the signal at δ 1.30 pm (δ OH) and the simplification of the signal at δ 4.16 from a

Table 3

Valency angles (Å) for compound II with e.s.d.s in parenthesis.

C(3)–Sn–C(7)	107.4(7)	C(3)–Sn–C(13)	120.6(6)
C(3)–Sn–C(19)	105.2(7)	C(7)–Sn–C(13)	106.8(5)
C(7)–Sn–C(19)	108.9(5)	C(13)–Sn–C(19)	107.5(5)
C(1)–O(1)–C(32)	114.0(18)	C(4)–O(4)–C(25)	109.7(14)
C(1)–O(5)–C(5)	110.5(14)	C(6)–O(6)–C(25)	112.3(16)
O(1)–C(1)–O(5)	113.3(21)	O(1)–C(1)–C(2)	108.9(17)
O(5)–C(1)–C(2)	111.8(16)	O(2)–C(2)–C(1)	107.8(16)
O(2)–C(2)–C(3)	111.8(16)	C(1)–C(2)–C(3)	110.6(17)
Sn–C(3)–C(2)	114.3(12)	Sn–C(3)–C(4)	114.0(13)
C(2)–C(3)–C(4)	108.1(15)	O(4)–C(4)–C(3)	111.0(15)
O(4)–C(4)–C(5)	107.5(15)	C(3)–C(4)–C(5)	113.5(16)
O(5)–C(5)–C(4)	111.4(17)	O(5)–C(5)–C(6)	110.8(16)
C(4)–C(5)–C(6)	110.2(17)	O(6)–C(6)–C(6)	108.1(16)
Sn–C(7)–C(8)	120.0(8)	Sn–C(7)–C(12)	120.1(10)
C(8)–C(7)–C(12)	120.0(12)	C(7)–C(8)–C(9)	120.0(11)
C(8)–C(9)–C(10)	120.0(12)	C(9)–C(10)–C(11)	120.0(12)
C(10)–C(11)–C(12)	120.0(11)	C(7)–C(12)–C(11)	120.0(12)
Sn–C(13)–C(14)	117.1(9)	Sn–C(13)–C(18)	122.9(9)
C(14)–C(13)–C(18)	120.0(11)	Sn–C(19)–C(24)	121.1(10)
C(14)–C(15)–C(16)	120.0(11)	O(4)–C(25)–C(26)	108.9(16)
C(16)–C(17)–C(18)	120.0(11)		
Sn–C(19)–C(20)	118.8(10)		
O(4)–C(25)–O(6)	113.5(16)		
O(6)–C(25)–O(26)	108.7(16)		
O(1)–Sn–C(3)	63.1(6)	O(4)–Sn–C(3)	49.0(6)
O(1)–Sn–C(13)	72.9(6)	O(4)–Sn–C(13)	92.6(4)
O(1)–Sn–C(7)	85.6(5)	O(4)–Sn–C(7)	155.8(5)
O(1)–Sn–C(19)	164.2(5)	O(4)–Sn–C(19)	77.6(5)
O(1)–Sn–O(4)	86.6(4)		

Table 4

^1H and ^{13}C NMR spectra of compound II (δ rel. to Me_4Si: J in Hz)

^1H NMR	H-1 [$J(H_1-H_2)$]	H-2 [$J(H_2-H_3)$] [[$J(^{119}Sn-^1H)$]]	H-3 [$J(H_3-H_4)$] [[$J(^{119}Sn-H)$]]	H-4 [$J(H_4-H_5)$] [[$J(^{119}Sn-H)$]]	H-5 [$J(H_5-H_{6'})$]	H-6	H-6′	OMe	OH [$J(H_2-OH)$]	PhCH	Aryl	Ref.
360 MHz: $CDCl_3$	4.66	4.28	2.79	4.53	4.07-4.03	3.87-3.77		3.08	2.04 [b]	5.65	7.61-7.57: *o*-Ph_3Sn [c]	This study
	[0]	[1.8]	[5.9]	[8.8]	[m] [a]	[m] [a]			[6.2]		7.36-7.30: *m-p*-Ph_3Sn	
		[[28.6]]	[[60]]	[[*ca.* 30]]							7.25-7.20 *Ph*CH	
270 MHz: C_6D_6	4.37	1.42 [d]	2.74	4.45	3.97	3.55	3.91	2.81	4.16 [d]	5.43	7.83-7.20	[6]
	[0]	[2.1]	[5.9]	[8.8]	[4.8]	[11.4]	[11.8]		[3.6]			
^{13}C NMR	C-1 [$J(^{119}Sn-^{13}C)$]	C-2 [$J(^{119}Sn-^{13}C)$]	C-3 [$J(^{119}Sn-^{13}C)$]	C-4 [$J(^{119}Sn-^{13}C)$]	C-5 [$J(^{119}Sn-^{13}C)$]	C-6 –	OMe	PhCH	Aryl [$J(^{119}Sn-^{13}C)$]			
90 MHz: $CDCl_3$	100.2 [e,f]	63.7 [g]	34.8	70.8 [g]	76.8 [g]	69.1	54.1	100.9 [e]	128.3, 128.1			This study
		[15.2]	[379]	[12.7]	[ca. 30]				127.8: *Ph*CH			
									139.6[519]*i*-C-Ph_3Sn			
									137.4[37]*m*-C-Ph_3Sn			
20 MHz: C_6H_6	101.1	70.8 [g]	35.3	72.2 [g]	64.3 [g]	69.4	53.7		128.4[11]*o*-C-Ph_3			
	[32.8]	[10.1]	[392]	[32]	[15.7]				125.8[0]*p*-C-Ph_3Sn			
								100.6			[6]	

[a] Unresolved multiplet. [b] Removed on D_2O shake. [c] $J(^{119}Sn-^1H)$ ca. 50 Hz. [d] We believe that assignments should be interchanged. [e] Could be interchanged. [f] Coupling content not determined. [g] Disagreement is assignment between the two studies.

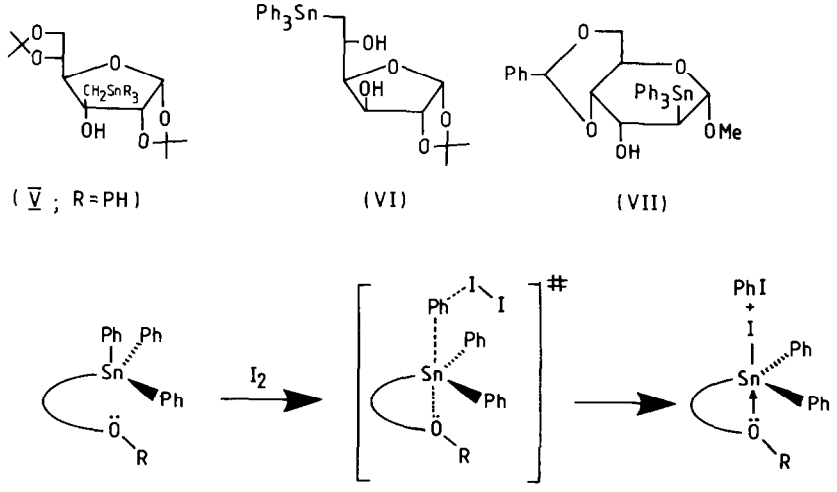
double of doublets to a doublet. The ^{13}C NMR spectra in $CDCl_3$ (this work) and in C_6D_6 [6] are also very similar (Table 2); however here again we disagree with some of the assignments previously made [6]; e.g. those for C_2, C_4 and C_5. Our assignments for these atoms are essentially based on values of the appropriate $J(^{119}Sn-^{13}C)$ coupling constants.

Solution and solid state conformations can of course be different. However, we do not believe, that even if II exists mainly as the skew conformation in solution, it does so as a consequence of the bulk of the Ph_3Sn unit. The solid-state structure shows that the Ph_3Sn group can be readily accommodated on the sugar cycle in the more favoured $^4C_1$ conformation.

Reactivities. Compound II is decomposed by CF_3CO_2H (1 equivalent); the only identified products being PhCHO and C_6H_6 (in a 1 : 2 mole ratio). The benzaldehyde clearly arises from the benzylidene protecting group and the two moles of benzene from protonolysis of two Ph–Sn bonds, despite the use of only 1 mole of the acid. The second protonolysis probably results from an intramolecular reaction involving a hydoxyl group.

The reactions with other electrophilic reagents, (E–Nu), such as I_2, Br_2 or $[Cl_2PtCOD]$ (COD = cycloocta-1,5-diene), are simpler in forming Ph–E (E = I, Br or ClPt(COD)) and the appropriate $NuPh_2Sn$-carbohydrate derivatives (Nu = I, Br or Cl).

In an earlier study [2], the relative reactivities of II and other stannyl-carbohydrates, including V (R = Ph), VI and VII, towards iodine were determined: values obtained were 0.5 : 1 : 1.9 : < 0.003 for II, V (R = Ph), VI and VII respectively. All four stannyl-carbohydrates have hydroxyl groups in β-positions to the tin centres. In all cases, reaction leads only to phenyl–tin cleavage. One factor, which would lead to a higher reactivity would be nucleophilic assistance by a neighbouring oxygen group in the transition state of the cleavage step [20]. For maximum effect, the participating nucleophilic (or donor) group should be *trans* coaxial to the cleaved phenyl–tin bond (Scheme 2). For VI, the β-HO- and Ph_3Sn- groups can readily achieve this situation, being in an acyclic chain; for V, the β-HO group is



Scheme 2.

also ideally sited to participate [see for example the structure of I]. We now have shown that in II, oxygen groups (but not the β-HO group on C-2) are placed in suitable positions for nucleophilic assistance. For VII, no groups appear able to aid the cleavage reaction and hence its rate of reaction with I_2 is much less than those for II, V (R=Ph) and VI.

Experimental

Methyl α-D-glucopyranoside [21], methyl-4,6-*O*-benzylidene-α-D-glucopyranoside [22], methyl 4,6-*O*-benzylidene-2-*O*-*p*-toluenesulphonyl-α-D-glycopyranoside [23]and methyl 2,3-anhydro-4,6-*O*-benzylidene-α-D-mannopyranoside [23] were obtained by published procedures.

Methyl 4,6-O-benzylidene-3-deoxy-3-triphenylstannyl-α-D-altropyranoside

A solution of triphenylstannyl-lithium (prepared from triphenyltin chloride (2.50 g, 6.50×10^{-3} mol) and lithium (0.46 g, 0.065 mol)) in dry THF was added slowly under a nitrogen atmosphere to a solution of methyl 2,3-anhydro-4,6-*O*-benzylidene − α-D-mannopyranoside (1.00 g, 3.80×10^{3} mol) in dry THF (25 ml). The green colour of the triphenylstannyl-lithium changed immediately on addition to give a brown solution. The reaction mixture was stirred for 1 h, hydrolysed with water (200 ml), neutralized with ammonium chloride, extracted into chloroform (3×50 ml) and dried over magnesium sulphate. The solvent was removed by rotary evaporation to leave a syrup which was taken up in cold diethyl ether and the bulk of the hexaphenylditin removed by filtration. The product was isolated by use of a Chromatron as a solid foam (eluent; diethyl ether : hexane 1 : 1). Yield 0.76 g, 33%. Recrystallised from EtOH, m.p. 165–167 °C (lit. [6] 166–168 °C).

Analysis: Found: C, 62.3; H, 5.2. $C_{32}H_{32}O_5Sn$ calcd.: C, 62.5; H, 5.2%. ^1H and ^{13}C NMR spectra are given in Table 4. ^{119}Sn NMR ($CDCl_3$, 134 MHz): δ −118.35 ppm (rel. to Me_4Sn). Mass spectrum (20 eV): *m/z* (%, fragment): 616(1, M^+), 539(4, $M^+ - Ph$), 507(2, $M^+ - Ph - OMe - H$), 479(3, $M^+ - PhCHO - OMe$), 478(3, $M^+ - PhCHO - OMe - H$), 457(4), 448(2), 433(1, $M^+ - Ph - PhCHO$), 401(3, $M^+ - Ph - PhCHO - OMe - H$), 395(2), 371(7), 351(100, Ph_3Sn^+), 291(30, Ph_2Sn^+OH), 274(13, Ph_2Sn^+), 197(29, $PhSn^+$), 149(91), 120(19, Sn^+), 105(29, PhCO).

Reactions

With iodine. To a solution of II (30.4 mg, 4.94×10^{-5} mol) in CCl_4 (4 ml) was added iodine (11.4 mg, 4.50×10^{-5} mol) in CCl_4 solution (0.45 ml) and the mixture kept in the dark until decolourization was complete. Analysis by GLC, using PhBr as an internal standard, indicated 102% formation of PhI. The solvent and all volatiles were removed from the reaction mixture under vacuum; the residue was taken up in CCl_4.

^1H NMR ($CDCl_4$, 220 MHz): 7.6–7.2 (m, 15H, Ph_2Sn + *Ph*CH), 5.64 (s, 1H, PhC*H*), 4.6–4.5 (m, 2H, H-1 + H-4), 4.30 (brs, 1H, H-2), 4.05–3.90 (m, 1H, H-5), 3.75–3.50 (m, 2H, H-6 + H-6′), 3.01 (s, 3H, OMe), 2.85 (brd, 1H, H-3), 2.80–2.65 (brs, 1H, OH).

With trifluoroacetic acid. To a solution of II (30.0 mg, 4.88×10^{-5} mol) in CCl_4 (4 ml) was added CF_3CO_2H (3.75 μl, 4.88×10^{-5} mol). The solution darkened in

colour and a black precipitate settled out. The ^{1}H NMR spectrum of the supernatant solution indicated the presence of PhH (δ 7.25) and PhCHO [δ 9.88 (s, 1H, 9.88); 7.92 (d, 2H), 7.67 (t, 1H) and 7.53 (t, 1H)].

Crystal structure determination of II

Crystal data. $C_{32}H_{32}O_5Sn$, $M = 616.29$, orthorhombic, $P2_12_12_1$, $a = 9.84(2)$, $b = 13.73(5)$, $c = 21.20(2)$ Å, $Z = 4$, $V = 2861(12)$ Å^3, $D_c = 1.39$ g cm^{-3}, $D_m = 1.41$ g cm^{-3}, $F(000) = 1256$, μ (Mo-K_α) = 9.3 cm^{-1}, $\lambda = 0.71069$ Å, T = room temperature.

Data collection and processing. Colourless crystal, 0.2 × 0.3 × 0.5 mm. Data were collected on a Nicolet P3 automated diffractometer, using monochromated Mo-K_α radiation. Unique intensities was measured with $2\theta < 50°$ as θ–2θ scans. A total of 2880 reflexions was measured of which 1280 had $F > 4\sigma(F)$ and were used for subsequent analysis. Range of *hkl*: $0 \leqslant h < 13$, $0 \leqslant k < 18$, $0 \leqslant l < 27$. Data were corrected for Lorentz, polarisation and absorption effects. Two reference reflexions, monitored periodically, showed no significant variation in intensity.

Structure analysis and refinement. The position of the tin atom was located from the three-dimensional Patterson function while the remaining non-hydrogen atoms were located from successive difference Fourier maps, using SHELX-76 [24]. The phenyl rings attached to tin were treated as regular hexagons and were subsequently refined as rigid groups. Hydrogen atoms were located and, with the exception of methyl hydrogens, were given ideal geometries. Coordinates of methyl hydrogens were calculated in idealized positions and subsequently, the entire methyl group was treated as a rigid unit and rotated to best fit the data. Full-matrix least-squares calculations on F with anisotropic thermal parameters for the tin, oxygen and carbon (C1–C6 and C25–C32) and isotropic thermal parameters for hydrogen and other carbon atoms converged at R 0.0617, R_w 0.0570 (for the structure shown in Fig. 2). The structure with the inverted configuration has higher R values. Atomic scattering factors were from SHELX-76. Final $w = 1.3873/[\sigma^2(F_o) + 0.001(F_o)^2]$, final $\Delta\rho_{min} = -0.30$, final $\Delta\rho_{max} = 0.44$ eA^{-3}. Molecular geometries were generated by the GX package [25].

Tables of anisotropic thermal parameters, H atoms positions, torsional angles, and structive factors are available from the authors.

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