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Synthesis, Characterization and Crystal Structure of (*Z*)-1-[2-(Tri-*o*-tolylstannyl)vinyl]-1-cyclopentanol and Its *o*-Tolylhalostannyl Derivatives

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(*Z*)-1-[2-(Tri-*o*-tolylstannyl)vinyl]-1-cyclopentanol (**1**) was synthesized by the additive reaction of 1-ethynylcyclopentanol with tri-*o*-tolyltin hydride. One or two of the *o*-tolyl groups of compound **1** was substituted by I, Br or Cl to yield derivatives of the type $\overline{CH_2(CH_2)_3}CH(OH)CH=CHSn(o\text{-tol})_{3-n}X_n$ [n = 1, X = I (**2**), Br (**3**), Cl (**4**); n = 2, X = Br (**5**)]. The compounds **1**–**5** were characterized by elemental analysis, ^{1}H NMR and FT-IR spectroscopy. The crystal and molecular structures of **1** and **2** have been determined by single crystal X-ray diffraction analysis. The Sn atom in **1** exhibits a tetrahedral geometry distorted towards trigonal bipyramid, due to a weak intramolecular interaction between Sn and hydroxyl O atoms [2.813(4) Å], while the Sn atom in **2** adopts a trigonal bipyramidal geometry with a significant Sn(1)←O(1) interaction [2.553(4) Å].

Key words: organotin compounds, aryltin compounds, FT-IR spectroscopy, NMR spectroscopy, crystal structure

Organotin compounds have been widely used in catalysis, organic synthesis, PVC stabilizer, timber antisepsis, pesticide and antiseptic dope *etc* [1–3]. In particular, some organotin compounds have been found to have good cytotoxic activity, which could be over 100 times of the *cis*-platinum complexes [4–7]. Therefore, the influence of the structural features of organotin compounds on the cytotoxicity has received considerable attention. The synthesis and cytotoxic activity of organotin compounds of the type (*Z*)-(Ar_3Sn)-CH=CH-C(OH)RR' (Ar = phenyl and *p*-tolyl) and their arylhalostannyl derivatives have been an active research subject [8–18]. The solid-state structures of (*Z*)-(Ar_3Sn)-CH=CH-C(OH)RR' exhibit a weak intramolecular HO→Sn interaction. The Sn atom in these compounds is located in a distorted tetrahedral geometry, while the Sn atom adopts a trigonal bipyramidal geometry in the diarylhalostannyl and aryldihalostannyl derivatives of (*Z*)-($Ar_{3-n}X_nSn$)-CH=CH-C(OH)RR' due to a strong HO→Sn interaction. The cytotoxic activity of these compounds is obviously related to the strength HO→Sn interaction, which is determined by the number and nature of the aryl and the Lewis

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acidities of the central tin atom [11,17,18], Effects of the phenyl and *p*-tolyl groups on both the structure and the cytotoxicity of this type of compounds have been well studied in previous work. However, no *o*-tolyl substituted organotin compound of this type has been reported. In this paper, the synthesis and structure of (*Z*)-1-[2-(tri-*o*-tolylstannyl)vinyl]-1-cyclopentanol (**1**) and it's *o*-tolyhalostannyl derivatives were reported. Their structural features, particularly the HO→Sn coordination interaction, are discussed. These compounds are possible to serve as a new model for further investigation on the structure-cytotoxicity relationship.

EXPERIMENTAL

General comments: Elemental analyses were carried out on a Perkin-Elmer PE 2400 CHN instrument. The ^{1}H NMR spectra were recorded in $CDCl_3$ on a Varian Mercury 300 MHz spectrometer. Infrared spectra (KBr pellets) were recorded on an Alpha Centauri FI/IR spectrometer (400–4000 cm^{-1} range). Cyclopentanone, tri-*o*-tolyltin chloride, $LiAlH_4$, iodonium chlorine, bromine and iodine were obtained from commercial source and used without further purification. 1-Ethynylcyclopentanol was prepared by a modified literature method [19]. Tri-*o*-tolyltin hydride was obtained from the reaction of tri-*o*-tolyltin chloride with $LiAlH_4$ in dried ether [20]. Ethyl ether was dried and distilled from Na-K alloy under nitrogen. Other solvents were used without purification.

*Syntheses of (Z)-1-[2-(tri-o-tolylstannyl)vinyl]-1-cyclopentanol **1***: Tri-*o*-tolyltin hydride (39.31 g, 100 mmol), 1-ethynylcyclopentanol (11.02 g, 100 mmol) and dibenzoyl peroxide (100 mg) were dissolved in 100 mL dry diethyl ether. The mixture was stirred under nitrogen for 40 h at room temperature and the solvent was evaporated off. The residue was crystallized from ethanol to yield 30.81 g (61.22%) of **1** as white crystalline solid. Single crystals suitable for X-ray analysis were obtained by slow evaporation of ethanol solution at room temperature over one week. M.p. 111.7~112.5°C. IR (KBr): ν_{C-O} 1053, ν_{O-H} 3572 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ: 0.75 (s, OH); 6.24 (d, J_{HH} = 12.6 Hz, CH-Sn); 6.79 (d, J_{HH} = 12.6 Hz, CH); 7.51 (d, J_{HH} = 6.9 Hz, *o*-H of Ph); 7.11–7.26 (m, *m*, *p*-H of Ph); 2.28 (s, Ph-CH_3).

*Syntheses of (Z)-1-[2-(iododi-o-tolylstannyl)vinyl]-1-cyclopentanol **2***: Iodine (0.76 g, 3.0 mmol) dissolved in 50 mL of CCl_4 was added dropwise with stirring to a solution of **1** (1.51 g, 3.0 mmol) in 20 mL of CCl_4 at room temperature. The color of iodine disappeared immediately. The mixture was stirred for 2 h and the solvent was then evaporated off. The residue was recrystallized from cyclohexane to yield 1.18 g (72.97%) of compound **2** as white crystals. Single crystals for X-ray analysis were obtained by slow evaporation of cyclohexane solution at room temperature over one week. M.p. 123.0~124.8°C. IR (KBr): ν_{C-O} 1032, ν_{O-H} 3566 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ: 1.88 (s, OH); 6.56 (d, J_{HH} = 11.7 Hz, CH-Sn); 6.68 (d, J_{HH} = 11.7 Hz, CH); 7.49 (d, J_{HH} = 7.5 Hz, *o*-H of Ph); 7.18–7.30 (m, *m*, *p*-H of Ph); 2.60 (s, Ph-CH_3).

*Syntheses of (Z)-1-[2-(bromodi-o-tolylstannyl)vinyl]-1-cyclopentanol **3***: Bromine (0.48 g, 3.0 mmol) in 10 mL of CCl_4 was added slowly with stirring to a solution of **1** (1.51 g, 3.0 mmol) in 20 mL of CCl_4 at –5°C. The colour of bromine disappeared immediately. The mixture was allowed to warm to room temperature and stirred for 1.5 h. The solvent was evaporated off and the residue was recrystallized from cyclohexane to give 1.01 g (68.43%) of compound **3** as white crystals. M.p. 155.9~155.6°C. IR (KBr): ν_{C-O} 1054, ν_{O-H} 3415 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ: 2.37 (s, OH); 6.52 (d, J_{HH} = 10.6 Hz, CH-Sn); 6.80 (d, J_{HH} = 10.6 Hz, CH); 7.71 (d, J_{HH} = 6.6 Hz, *o*-H of Ph); 7.22–7.37 (m, *m*, *p*-H of Ph); 2.45 (s, Ph-CH_3).

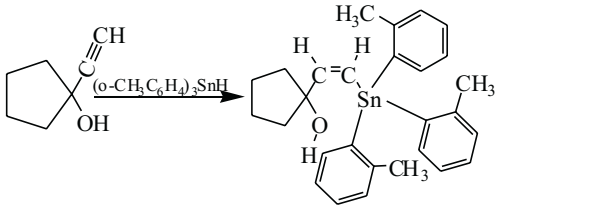
*Syntheses of (Z)-1-[2-(chlorodi-o-tolylstannyl)vinyl]-1-cyclopentanol **4***: A solution of ICl (0.81 g, 5.0 mmol) in 20 mL of CCl_4 was added dropwise with stirring to a solution of **1** (2.52 g, 5.0 mmol) in 30 mL of CCl_4 at 0°C. The color of ICl disappeared immediately. The mixture was allowed to warm to room temperature and stirred for 1 h. The solvent was evaporated off and the residue was recrystallized two times from cyclohexane to yield 1.90 g (84.90%) of compound **4** as white needles. M.p. 131.7~132.5°C. IR (KBr): ν_{C-O} 1060, ν_{O-H} 3390 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ: 2.69 (s, OH); 6.40 (d, J_{HH} = 10.4 Hz, CH-Sn); 6.85 (d, J_{HH} = 10.4 Hz, CH); 7.49, (d, J_{HH} = 6.7 Hz, *o*-H of Ph); 7.08–7.40 (m, *m*, *p*-H of Ph); 2.45 (s, Ph-CH_3).

Syntheses of (Z)-1-[2-(dibromo-o-tolylstannyl)vinyl]-1-cyclopentanol ***5***: A solution of bromine (0.96 g, 6.0 mmol) in 20 mL of CCl_4 was added dropwise with stirring to a solution of **1** (1.51 g, 3.0 mmol) in 30 mL of CCl_4 at –5°C. After the solution became almost colourless, the solvent was evaporated off and the residue was recrystallized from $HCCl_3$/cyclohexane (1:3 by volume) to yield 1.12 g (77.67%) of compound **5** as white crystals. M.p. 123.2~124.1°C. IR (KBr): ν_{C-O} 1054, ν_{O-H} 3331 cm^{-1}. ^1H NMR ($CDCl_3$): δ: 2.88 (s, OH); 6.54 (d, J_{HH} = 10.2 Hz, CH-Sn); 6.73 (d, J_{HH} = 10.2 Hz, CH); 7.52 (d, J_{HH} = 6.9 Hz, *o*-H of Ph); 7.21–7.32 (m, *m*, *p*-H of Ph); 2.53 (s, Ph-CH_3).

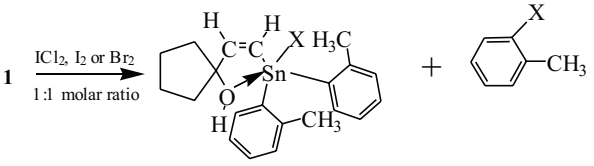
Crystal structure determinations of compound ***1*** *and* ***2***: Diffraction data of compound **1** and **2** were collected on a Rigaku RAXIS-RAPID diffractometer at room temperature, with graphite monochromated Mo-Kα radiation (λ = 0.71073 Å) by the ω scan mode. Empirical absorption corrections (multi-scan) were applied in case. The structures were solved by the heavy-atom method (SHELXTL-97) and refined by full-matrix least-squares on F^2 (SHELXTL-97) [21]. All non-hydrogen atoms were refined anisotropically, and the positions of hydrogen atoms on carbon atoms were calculated theoretically.

RESULTS AND DISCUSSION

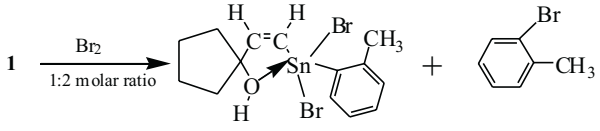
Synthesis: Compounds **1**–**5** were obtained from reactions shown in Scheme 1. **1** was synthesized by addition of the corresponding tri-*o*-tolyltin hydride to the triple bond of 1-ethynylcyclopentanol. Reactions of compound **1** with halogens in a 1:1 or 1:2 molar ratio yield the corresponding mono- and dihalides, respectively. During the synthesis of compound **1**, a small amount of dibenzoyl peroxide was added to the reaction mixture as evocating agent and the reaction was carried out under nitrogen.



compound **1**



X = I compound **2**
X = Br compound **3**
X = Cl compound **4**



compound **5**

Scheme 1. Synthetic routes to compounds **1**–**5**.

The reaction has been tried in different solvents such as THF, benzene and toluene, and it was found that **1** could be obtained in higher yield from the reaction carried out in diethyl ether. When pure tri-*o*-tolyltin hydride was used instead of the one formed *in situ* by the reaction of tri-*o*-tolyltin chloride with $LiAlH_4$, the reaction went to completion in less time and **1** was produced in better yield. In addition, hexa-*o*-tolylditin was observed as an insoluble by-product in these reactions.

Compound **1** reacts with I_2, Br_2 or ICl in carbon tetrachloride or chloroform to produce monohalides **2**–**4**, but the yields of **2**–**4** produced in chloroform are lower. It was found that the reactivity of halogens is in the order $ICl > Br_2 > I_2$. The fact that **2**–**4** were obtained in high yields indicates that the addition reaction of the C=C double bond of the vinyl group in **1** with halogen is less reactive than the substituent reaction of the *o*-tolyl group by halogen atom. Pure **2**–**4** was obtained by recrystallization from cyclohexane. In order to isolate the product in high yield, *o*-toluene halide formed in the reaction as a by-product should be removed together with the reaction solvent before recrystallization. Compound **5** was synthesized in two ways. One is from the reaction of **1** with bromine in 1:2 molar ratio. The other way is from the reaction of **3** with bromine in 1:1 molar ratio. Higher yield was obtained from the latter reaction. Attempts to make tribromide derivative by substituting all of the three *o*-tolyl groups in **1** with bromine atoms were not successful. When the reaction of **1** with bromine was carried out in 1:3 molar ratio, the color of bromine did not disappeared after the reaction mixture was stirred for 5 h and the dibromide derivative **5** was obtained as major product. Pure **5** was isolated by recrystallization from mixed solvent of $CHCl_3$/cyclohexane (1:3 by volume). Compounds **1**–**5** were characterized by IR, ^1H NMR and elemental analysis.

*Description of compounds **1** and **2***: The molecular structures of compound **1** and **2** are given in Fig. 1 and Fig. 2. The crystal parameters and procedure information corresponding to data collection and structure refinement are given in Table 1.

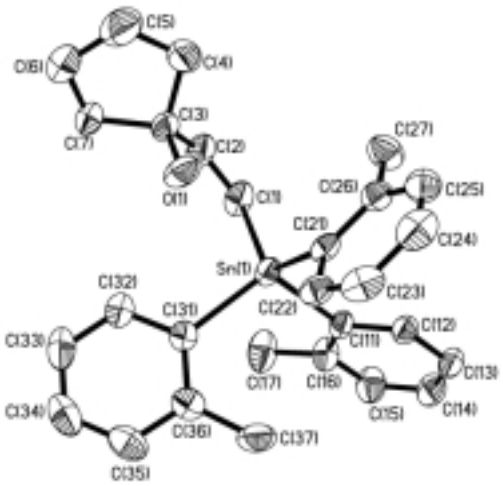


Figure 1. Molecular structure of compound **1**, showing the atomic numbering scheme.

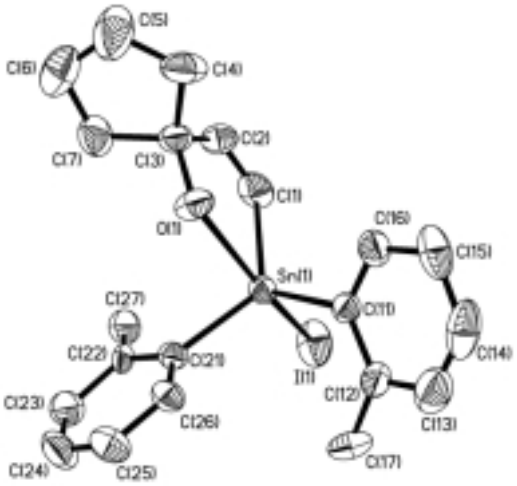


Figure 2. Molecular structure of compound **2**, showing the atomic numbering scheme.

The selected distances and bond angles are listed in Table 2. The Sn atom in **1**, bonded to three *o*-tolyl groups and the C(1) atom of the vinyl residue, adopts a distorted tetrahedral geometry with C–Sn–C angles ranging from 100.67(11)° to 120.83(14)°. The C(1)–Sn(1)–C(21) angle (120.83(14)°) is significantly larger than other C–Sn–C angles due to weak coordination of the O(1) atom of the cyclopentanol hydroxyl group. The distance between the O(1) and the Sn(1) atoms is 2.813(4) Å, which is significantly less than the sum of their van der Waals radii [3.70 Å] [22]). The weak coordination of the O(1) atom also influences the strength of the Sn(1)–C(*o*-tolyl).

Table 1. Crystal data and details of structure refinement parameters for compounds **1** and **2**.

Compound	**1**	**2**
Formula	$C_{28}H_{32}OSn$	$C_{21}H_{25}IOSn$
Molecular weight	503.23	539.00
Temperature (K)	293(2)	293(2)
Crystal size	0.48×0.30×0.26	0.64×0.21×0.05
Crystal color	colorless	colorless
Crystal system	monoclinic	monoclinic
Space group	*P*2(1)/*n*	*P*2(1)/*c*
Cell constants		
a (Å)	10.496(2)	10.234(2)
b (Å)	17.658(4)	10.198(2)
c (Å)	13.193(3)	20.479(4)
β(°)	90.60(3)	101.13(3)
V (Å^3)	2445.1(8)	2097.1(7)
Z	4	4
D_{calc} (g cm^{-3})	1.367	1.707
F (000)	1032	1048
Scan mode	ω	ω
$2\theta_{max}$	54.72	54.96
Total reflections collected	5426	4702
Absorption coefficient μ(Mo Kα)(mm^{-1})	1.061	2.695
R_1 (on *F* for reflections with $I>2\sigma(I)$)	0.0323 (for 3977 reflections)	0.0432 (for 2678 reflections)
wR_2 (on F^2 for all reflections)	0.0948 (for 5426 reflections)	0.1268 (for 4702 reflections)
Goodness of fit	0.981	0.967

Table 2. Selected bond distances (Å) and angles (°) in **1** and **2**.

Compound **1**		Compound **2**	
Sn(1)-C(1)	2.118(3)	Sn(1)-C(1)	2.117(8)
Sn(1)-C(21)	2.150(3)	Sn(1)-C(21)	2.137(6)
Sn(1)-C(31)	2.137(3)	Sn(1)-C(11)	2.148(6)
Sn(1)-C(11)	2.199(3)	Sn(1)-I(1)	2.7849(8)
Sn(1)-O(1)	2.813(4)	Sn(1)-O(1)	2.553(4)
O(1)-C(3)	1.402(4)	O(1)-C(3)	1.453(7)
C(1)-C(2)	1.294(5)	C(1)-C(2)	1.314(11)
C(2)-C(3)	1.511(5)	C(2)-C(3)	1.446(11)
C(1)-Sn(1)-C(31)	108.18(13)	C(1)-Sn(1)-C(21)	123.8(3)
C(1)-Sn(1)-C(21)	120.83(14)	C(1)-Sn(1)-C(11)	116.1(3)
C(31)-Sn(1)-C(21)	112.78(11)	C(21)-Sn(1)-C(11)	110.6(2)
C(1)-Sn(1)-C(11)	104.15(13)	C(1)-Sn(1)-I(1)	95.1(2)
C(31)-Sn(1)-C(11)	109.13(12)	C(21)-Sn(1)-I(1)	102.52(16)
C(21)-Sn(1)-C(11)	100.67(11)	C(11)-Sn(1)-I(1)	103.91(16)
C(11)-Sn(1)-O(1)	166.5(5)	C(1)-Sn(1)-O(1)	68.7(2)
C(2)-C(1)-Sn(1)	127.9(3)	C(11)-Sn(1)-O(1)	90.18 (19)
C(1)-C(2)-C(3)	128.5(3)	C(21)-Sn(1)-O(1)	81.95(19)
O(1)-C(3)-C(4)	107.3(4)	I(1)-Sn(1)-O(1)	162.30(10)
O(1)-C(3)-C(2)	106.6(3)	Sn(1)-O(1)-C(3)	113.2(4)
		Sn(1)-C(1)-C(2)	123.3(6)
		C(1)-C(2) -C(3)	126.9(7)
		C(2) -C(3)-O(1)	105.6(5)

As a result, the Sn(1)–C(11) is longer by 0.04 Å than the other two Sn(1)–C(*o*-tolyl) bonds. The fact that the *Z* isomer rather than the *E* isomer was obtained from this type of reactions [11] might be attributed to the weak intramolecular O→Sn coordination.

The Sn atom in **2** is five coordinated and the molecule has a distorted trigonal bipyramid geometry with the trigonal plane defined by C(1), C(11) and C(21) atoms and the axial positions occupied by the I(1) and O(1) atoms, which is similar to the analogue (*Z*)-1-[2-(chlorodi-*p*-tolylstannyl)vinyl]-1-cycloheptanol [17]. The Sn···O(1) distance of 2.553(4) Å is in the range of a normal Sn–O coordination bond length [11,14,17], indicating that the Sn atom in **2** is formally coordinated by the O(1) atom of the hydroxyl group and the Lewis acidity of the Sn atom in the $(o\text{-tolyl})_2SnI$ moiety is greater than in the $(o\text{-tolyl})_3Sn$ moiety. On the other hand, the O(1)–C(3) bond (1.453(7) Å) in **2** is weaker than the corresponding bond (1.402(4) Å) in **1** due to the strong coordination interaction between the O(1) and the Sn atoms. The Sn(1)–C(1)–C(2) angle (123.3(6)°) in **2** is considerably smaller than the equivalent angle (127.9(3)°) in **1** and the C(1)–C(2) bond (1.314(11) Å) in **2** is longer than the corresponding one (1.294(5) Å) in **1**, which is apparently resulted from the strain of the five-membered ring.

^{1}H NMR and IR spectra: The solution ^{1}H NMR spectra of compounds **1**–**5** are consistent with their structures. All spectra show characteristic ethylenic proton signals of doublet of doublets with $^3J_{(HC=CH)}$ = 10.2–12.6 Hz in the regions of 6.28–6.60 and 6.64–6.85 ppm. As example, the vinyl and *o*-tolyl regions of ^{1}H NMR spectra of **1**, **2** and **5** are illustrated in Fig. 3. The $^3J_{(HC=CH)}$ coupling constant of 12.6 Hz observed for **1** is quite large since *cis* coupling constants in similar compounds with a five-membered ring substituent usually amount to 8 Hz or even less [23].

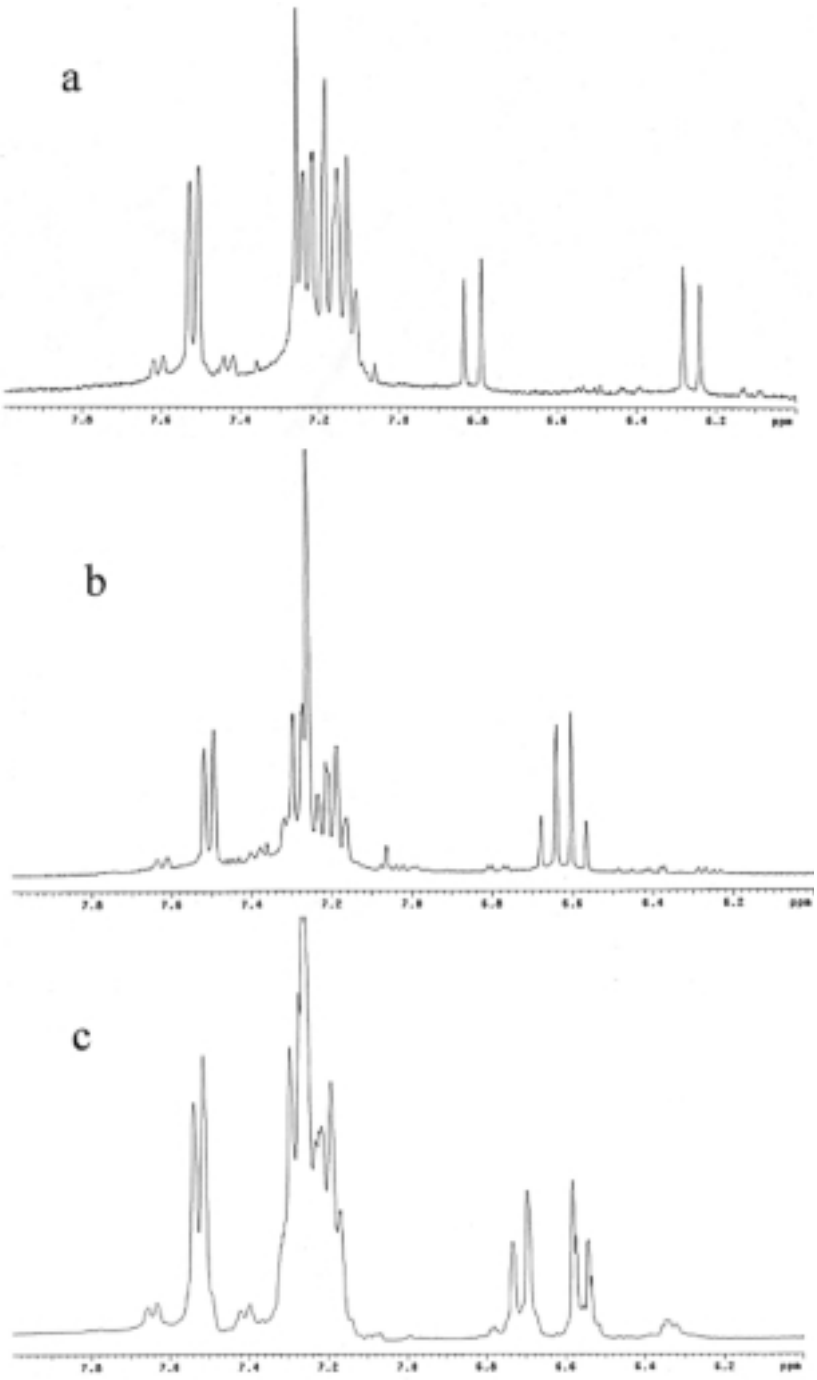


Figure 3. The signal patterns of ethylenic and *o*-tolyl proton of the compound **1**, **2** and **5**.
a. (*Z*)-1-[2-(tri-*o*-tolylstannyl)vinyl]-1-cyclopentanol **1**;
b. (*Z*)-1-[2-(iododi-*o*-tolylstannyl)vinyl]-1-cyclopentanol **2**;
c. (*Z*)-1-[2-(dibromo-*o*-tolylstannyl)vinyl]-1-cyclopentanol **5**.

In respect to the stannyl group, the $^3J_{(HC=CH)}$ coupling constant decreases in the order Sn(*o*-tolyl)$_3$ > Sn(*o*-tolyl)$_2$I > Sn(*o*-tolyl)$_2$Br > Sn(*o*-tolyl)$_2$Cl > Sn(*o*-tolyl)Br$_2$. The chemical shift of OH shifts to high field in the same order, which indicates that the strength of the HO→Sn interaction increases with the decrease in the steric hindrance of the stannyl group.

The infrared spectra of all compounds **1**–**5** show the presence of strong absorptions in the regions 1032–1054 and 3331–3572 cm^{-1}, which can be assigned to $\nu_{(C-O)}$ and $\nu_{(O-H)}$ stretching vibrations, respectively.

Supplementary material. Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 215199, Copies of this information may be obtained free of charge from The Director, CCDC 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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