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Synthesis, characterization and crystal structures of (*Z*)-1-(triarylstannyl)-3-phenyl-1-buten-3-ols and their arylhalostannyl derivatives

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Abstract

(*Z*)-1-(tri-*o*-tolylstannyl)-3-phenyl-1-buten-3-ol (**1**) and (*Z*)-1-(tri-*p*-tolylstannyl)-3-phenyl-1-buten-3-ol (**2**) were synthesized by the additive reaction of 3-phenyl-1-butyn-3-ol with tri-*o*-tolyltin and tri-*p*-tolyltin hydride. One of the aryl groups in compounds **1** and **2** was substituted by Cl, Br, I to yield derivatives of the type $PhC(CH_3)(OH)CH{=}CHSn(aryl)_2X$ [aryl = *o*-tolyl, X = Cl (**3**); aryl = *p*-tolyl, X = Cl (**4**); aryl = *o*-tolyl, X = Br (**5**); aryl = *p*-tolyl, X = Br (**6**); aryl = *o*-tolyl, X = I (**7**); aryl = *p*-tolyl, X = I (**8**)]. Compounds **1**–**8** were characterized by elemental analysis, ^1H NMR and FT-IR spectroscopy. The crystal structures of **1**, **2** and **5** have been determined by single crystal X-ray diffraction analysis. The Sn atom in **1** and **2** exhibits tetrahedral geometry distorted towards trigonal bipyramidal due to a weak intramolecular interaction between Sn and the hydroxyl O atoms [2.879(5) and 2.859(4) Å], while the Sn atom in **5** adopts a trigonal bipyramidal geometry with a significant Sn(1)←O interaction [2.483(4) Å].

Keywords: Organotin compounds; Aryltin compounds; FT-IR spectroscopy; NMR spectroscopy; X-ray crystal structure

1. Introduction

Since Crowe et al. [1] reported the antitumour activity and hypotoxicity of organotin compounds, great attention has been paid to the synthesis, structure and antitumour activity of such compounds. In particular, compounds of the type (*Z*)–(Ar_3Sn)–CH=CH–C(OH)RR′ (Ar = phenyl and *p*-tolyl) and their arylhalostannyl derivatives have been actively studied [2–12]. The solid-state structures of (*Z*)–(Ar_3Sn)–CH=CH–C(OH)RR′ exhibit a weak intramolecular HO → Sn interaction. As a result, the Sn atom in these compounds is located in a distorted tetrahedral environment, while the Sn atom adopts a trigonal bipyramidal geometry in the diarylhalostannyl and aryldihalostannyl derivatives of the type (*Z*)–($Ar_{3-n}X_nSn$)–CH=CH–C(OH)RR′ due to strong HO → Sn interaction. It has been reported that the antitumor activity of these compounds is related to the strength of the HO → Sn interaction, which is determined by the number and nature of the aryl groups and the Lewis acidity of the central tin atom [5,11,12]. In this paper, the synthesis and structure of (*Z*)-1-(triarylstannyl)-3-phenyl-1-buten-3-ols (aryl = *o*-tolyl and *p*-tolyl) and their arylhalostannyl derivatives are reported. Their structural features, particularly the HO → Sn coordination interaction, are discussed. These compounds are likely to serve as new models for further investigation on the structure-antitumor activity relationship.

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2. Experimental

2.1. Reagents and general techniques

Elemental analyses were carried out on a Perkin–Elmer PE 2400 CHN instrument and gravimetric analysis was carried out for Sn. ^{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).

Hypnone, tri-*o*-tolyltin chloride, tri-*p*-tolyltin chloride, $LiAlH_4$, iodine monochloride, bromine and iodine were obtained from commercial sources and used without further purification. 3-Phenyl-1-butyn-3-ol was prepared by a modified literature method [13]. Tri-*o*-tolyltin and tri-*p*-tolyltin hydrides were obtained from reaction of tri-*o*-tolyltin chloride and tri-*p*-tolyltin chloride with $LiAlH_4$ in dried diethyl ether [14,15]. Diethyl ether was dried and distilled from Na–K alloy under nitrogen. Other solvents were used without purification.

2.2. Synthesis

2.2.1. Synthesis of compounds **1** and **2**

3-Phenyl-1-butyn-3-ol (14.62 g, 100 mmol) and dibenzoyl peroxide (100 mg) were added to a solution of tri-*o*-tolyltin hydride in diethyl ether prepared from the reaction of tri-*o*-tolyltin chloride (53.44 g, 125 mmol) with $LiAlH_4$ (4.74 g, 125 mmol). The mixture was stirred for 30 h at room temperature under nitrogen and the solvent was evaporated off. The residue was recrystallized from ethanol to yield 45.30 g of **1** as a white crystalline solid.

The same procedure was used for the reaction of 3-phenyl-1-butyn-3-ol with tri-*p*-tolyltin hydride. Single crystals of both compounds suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution at room temperature over one week.

2.2.2. Reaction of compounds **1** and **2** with halogens

Bromine (160 mg, 1 mmol) in 15 ml of CCl_4 was added slowly with stirring to a solution of **1** (539 mg, 1 mmol) in 20 ml of CCl_4 at −5 °C. The color of bromine 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 from cyclohexane to give 375 mg of compound **5** as white crystals.

The other vinyl-*o*-tolyltin and vinyl-*p*-tolyltin halides were prepared analogously using ICl or I_2.

*2.2.2.1. (Z)-1-(tri-o-tolylstannyl)-3-phenyl-1-buten-3-ol (**1**).* Yield: 85%; m.p. (recrystallized from ethanol): 126.6–127.7 °C. IR (KBr pellets): ν_{CO} 1068 cm^{-1}, ν_{OH} 3552 cm^{-1}. ^{1}H NMR ($CDCl_3$, ppm): δ 1.54 (s, OH); 6.28 (d, $J_{HH} = 12.6$ Hz, CH–Sn); 7.07 (d, $J_{HH} = 12.6$ Hz, CH); 7.58 (d, $J_{HH} = 7.2$ Hz, *o*-H, Ph); 7.10–7.32 (m, *m*- + *p*-H, Ph); 2.27 (s, CH_3, Ph–CH_3); 1.52 (s, CH_3). *Anal.* Calc. for $C_{31}H_{32}OSn$: C, 69.04; H, 5.98; Sn, 22.20. Found: C, 68.95; H, 5.88; Sn, 22.09%.

*2.2.2.2. (Z)-1-(tri-p-tolylstannyl)-3-phenyl-1-buten-3-ol (**2**).* Yield: 72%; m.p. (recrystallized from ethanol): 97.2–98.0 °C. IR (KBr pellets): ν_{CO} 1066 cm^{-1}, ν_{OH}: 3550 cm^{-1}. ^{1}H NMR ($CDCl_3$, ppm): δ 1.76 (s, OH); 6.23 (d, $J_{HH} = 12.6$ Hz, CH–Sn); 7.05 (d, $J_{HH} = 12.6$ Hz, 1H, CH); 7.50 (d, $J_{HH} = 7.8$ Hz, *o*-H, Ph); 7.15–7.31 (m, *m*- + *p*-H, Ph); 2.34 (s, CH_3, Ph–CH_3); 1.61 (s, CH_3). *Anal.* Calc. for $C_{31}H_{32}OSn$: C, 69.04; H, 5.97; Sn, 22.20. Found: C, 68.92; H, 5.85; Sn, 22.19%.

*2.2.2.3. (Z)-1-(chlorodi-o-tolylstannyl)-3-phenyl-1-buten-3-ol (**3**).* Yield: 63%; m.p. (recrystallized from cyclohexane): 136.8–137.7 °C. IR (KBr pellets): ν_{CO} 1060 cm^{-1}, ν_{OH} 3390 cm^{-1}. ^{1}H NMR ($CDCl_3$, ppm): δ 2.69 (s, OH); 6.40 (d, $J_{HH} = 10.2$ Hz, CH–Sn); 6.86 (d, $J_{HH} = 10.2$ Hz, CH); 7.49, 7.61 (d, $J_{HH} = 6.7$ Hz, *o*-H, Ph); 7.08–7.40 (m, *m*- + *p*-H, Ph); 2.55 (s, CH_3, Ph–CH_3); 1.68 (s, CH_3). *Anal.* Calc. for $C_{24}H_{25}ClOSn$: C, 59.60; H, 5.21; Sn, 24.55. Found: C, 59.62; H, 5.30; Sn, 24.60%.

*2.2.2.4. (Z)-1-(chlorodi-p-tolylstannyl)-3-phenyl-1-buten-3-ol (**4**).* Yield: 71%; m.p. (recrystallized from cyclohexane): 133.6–134.8 °C. IR (KBr pellets): ν_{CO} 1055 cm^{-1}, ν_{OH} 3383 cm^{-1}. ^{1}H NMR ($CDCl_3$, ppm): δ: 2.70 (s, OH); 6.38 (d, $J_{HH} = 9.8$ Hz, CH–Sn); 6.83 (d, $J_{HH} = 9.8$ Hz, CH); 7.48 (d, $J_{HH} = 6.5$ Hz, *o*-H, Ph); 7.04–7.39 (m, *m*- + *p*-H, Ph); 2.56 (s, CH_3, Ph–CH_3); 1.75 (s, CH_3). *Anal.* Calc. for $C_{24}H_{25}ClOSn$: C, 59.60; H, 5.21; Sn, 24.55. Found: C, 59.63; H, 5.29; Sn, 24.65%.

*2.2.2.5. (Z)-1-(bromodi-o-tolylstannyl)-3-phenyl-1-buten-3-ol (**5**).* Yield: 71%; m.p. (recrystallized from cyclohexane): 135.3–136.1 °C. IR (KBr pellets): ν_{CO} 1060 cm^{-1}, ν_{OH} 3400 cm^{-1}. ^{1}H NMR ($CDCl_3$, ppm): δ 2.48 (s, OH); 6.48 (d, $J_{HH} = 11.4$ Hz, CH–Sn); 6.92 (d, $J_{HH} = 11.4$ Hz, CH); 7.51 (d, $J_{HH} = 6.9$ Hz, *o*-H, Ph); 7.03–7.37 (m, *m*- + *p*-H, Ph); 2.54 (s, CH_3, Ph–CH_3); 1.64 (s, CH_3). *Anal.* Calc. for $C_{24}H_{25}BrOSn$: C, 54.59; H, 4.77; Sn, 22.48. Found: C, 54.49; H, 4.81; Sn, 22.58%.

*2.2.2.6. (Z)-1-(bromodi-p-tolylstannyl)-3-phenyl-1-buten-3-ol (**6**).* Yield: 77%; m.p. (recrystallized from cyclohexane): 126.4–127.5 °C. IR (KBr pellets): ν_{CO} 1060 cm^{-1}, ν_{OH} 3392 cm^{-1}. ^{1}H NMR ($CDCl_3$, ppm): δ 2.52 (s, OH); 6.50 (d, $J_{HH} = 11.2$ Hz, CH–Sn); 6.94 (d, $J_{HH} = 11.2$ Hz, CH); 7.51 (d, $J_{HH} = 6.8$ Hz, *o*-H, Ph); 7.05–7.40 (m, m- + p-H, Ph); 2.55 (s, CH_3, Ph–CH_3); 1.68 (s, CH_3). *Anal.* Calc. for $C_{24}H_{25}BrOSn$: C, 54.59; H, 4.77; Sn, 22.48. Found: C, 54.50; H, 4.71; Sn, 22.53%.

2.2.2.7. (Z)-1-(iododi-o-tolylstannyl)-3-phenyl-1-buten-3-ol (7). Yield: 85%; m.p. (recrystallized from cyclohexane): 145.2–145.9 °C. IR (KBr pellets): ν_{CO} 1070 cm^{-1}, ν_{OH} 3405 cm^{-1}. ^1H NMR ($CDCl_3$, ppm): δ 2.38 (s, OH); 6.53 (d, $J_{HH} = 11.7$ Hz, CH–Sn); 6.80 (d, $J_{HH} = 11.7$ Hz, CH); 7.49 (d, $J_{HH} = 7.2$ Hz, *o*-H, Ph); 7.04–7.36 (m, *m*- + *p*-H, Ph); 2.53 (s, CH_3, Ph–CH_3); 1.62 (s, CH_3). *Anal.* Calc. for $C_{24}H_{25}IOSn$: C, 50.13; H, 4.38; Sn, 20.64. Found: C, 50.08; H, 4.31; Sn, 20.77%.

2.2.2.8. (Z)-1-(iododi-p-tolylstannyl)-3-phenyl-1-buten-3-ol (8). Yield: 81%; m.p. (recrystallized from cyclohexane):140.2–141.5 °C. IR (KBr pellets): ν_{CO} 1065 cm^{-1}, ν_{OH}: 3398 cm^{-1}. ^1H NMR ($CDCl_3$, ppm): δ 2.39 (s, OH); 6.55 (d, $J_{HH} = 11.5$ Hz, CH–Sn); 6.89 (d, $J_{HH} = 11.5$ Hz, CH); 7.52 (d, $J_{HH} = 7.2$ Hz, *o*-H, Ph); (m, *m*- + *p*-H, Ph); 2.52 (s, CH_3, Ph–CH_3); 1.64 (s, CH_3). *Anal.* Calc. for $C_{24}H_{25}IOSn$: C, 50.13; H, 4.38; Sn, 20.64. Found: C, 50.11; H, 4.45; Sn, 20.76%.

2.3. X-ray crystallography

Diffraction data for compounds **1**, **2** and **5** were collected on a Siemens P4 diffractometer with graphite monochromated Mo Kα radiation ($\lambda = 0.71073$ Å) at ambient temperature. The structures were solved by the heavy-atom method (SHELXS 97), and were refined by full-matrix least squares techniques (SHELXL 97) [16]. Non-hydrogen atoms were refined anisotropically. The hydrogen atoms were generated geometrically. The parameters of data collection and structure refinement are given in Table 1.

3. Results and discussion

3.1. Synthesis

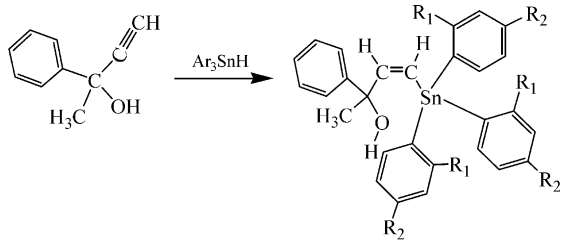
Compounds **1**–**8** were synthesized according to Scheme 1. Reactions of compounds **1** and **2** with halogens in a 1:1 molar ratio yield the corresponding monohalides **3**, **4**, **5**, **6**, **7** and **8**, respectively. Compounds **1**–**8** were all characterized by IR, ^1H NMR and elemental analysis.

3.2. ^1H NMR and IR spectra

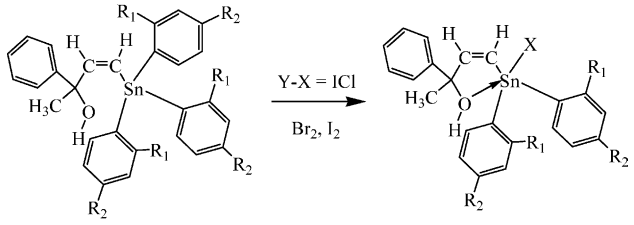
The solution ^1H NMR spectra of compounds **1**–**8** are consistent with their structures. All spectra show characteristic ethylenic proton signals of a doublet of doublets with $^3J_{(HC=CH)} = 9.8$–12.6 Hz in the regions of 6.27–6.57 and 6.80–7.07 ppm. The $^3J_{(HC=CH)}$ coupling constant of 12.6 Hz observed for **1** and **2** is quite large since the cis coupling constant in similar compounds with a five-membered ring substituent usually amounts to 8 Hz or even less [17]. In regard to the stannyl group, the $^3J_{(HC=CH)}$ coupling constant decreases in the order $Sn(aryl)_3 > Sn(aryl)_2I > Sn(aryl)_2Br > Sn(aryl)Cl$,

Table 1
Crystal data and details of structure refinement parameters for compounds **1**, **2** and **5**

Compound	**1**	**2**	**5**
Formula	$C_{31}H_{32}OSn$	$C_{31}H_{32}OSn$	$C_{24}H_{25}BrOSn$
Molecular weight	539.26	539.26	528.04
Temperature (K)	293(2)	293(2)	293(2)
Crystal size (mm)	$0.52 \times 0.44 \times 0.32$	$0.52 \times 0.40 \times 0.32$	$0.50 \times 0.46 \times 0.32$
Wavelength (Å)	0.71073	0.71073	0.71073
Crystal system	triclinic	monoclinic	monoclinic
Space group	$P\bar{1}$	$P2_1/n$	$P2_1/c$
Cell constants			
a (Å)	10.5044(19)	11.428(3)	17.879(2)
b (Å)	11.298(3)	18.840(4)	7.5287(13)
c (Å)	11.9927(18)	13.0874(16)	16.636(3)
α (°)	68.932(14)		
β (°)	86.898(13)	108.384(13)	92.071(14)
γ (°)	86.343(17)		
V (Å^3)	1324.7(4)	2674.0(9)	2237.8(6)
Z	2	4	4
$D_{calc.}$ (g cm^{-3})	1.352	1.340	1.567
$F(000)$	552	1104	1048
Scan mode	ω	ω	ω
$2\theta_{max}$	52.02	52.00	52.02
Absorption coefficient μ(Mo Kα) (mm^{-1})	0.984	0.975	2.938
R_1 (on F for reflections with $I > 2\sigma(I)$)	0.0498 (for 4189 reflections)	0.0344 (for 3423 reflections)	0.0434 (for 2849 reflections)
wR_2 (on F^2 for all reflections)	0.1332 (for 5071 reflections)	0.0713 (for 5249 reflections)	0.1004 (for 4405 reflections)
Goodness-of-fit	1.046	0.833	0.900



1: $R_1 = CH_3$, $R_2 = H$. **2**: $R_1 = H$, $R_2 = CH_3$,



3: $R_1 = CH_3$, $R_2 = H$, X = Cl. **4**: $R_1 = H$, $R_2 = CH_3$, X = Cl.

5: $R_1 = CH_3$, $R_2 = H$, X = Br. **6**: $R_1 = H$, $R_2 = CH_3$, X = Br.

7: $R_1 = CH_3$, $R_2 = H$, X = I. **8**: $R_1 = H$, $R_2 = CH_3$, X = I.

Scheme 1.

$Sn(o\text{-tolyl})_3 > Sn(p\text{-tolyl})_3$ and $Sn(o\text{-tolyl})_2X > Sn(p\text{-tolyl})_2X$. The chemical shifts of OH shift to high field following 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**–**8** show the presence of strong absorptions in the regions of 1055–1070 and 3398–3552 cm^{-1}, which can be assigned to $\nu_{(C-O)}$ and $\nu_{(O-H)}$ stretching vibrations, respectively.

3.3. Description of compounds *1*, *2* and *5*

The molecular structures of compounds **1**, **2** and **5** are given in Figs. 1–3, respectively. The Sn atom in **1**, bonded to three *o*-tolyl groups and the C(1) atom of the

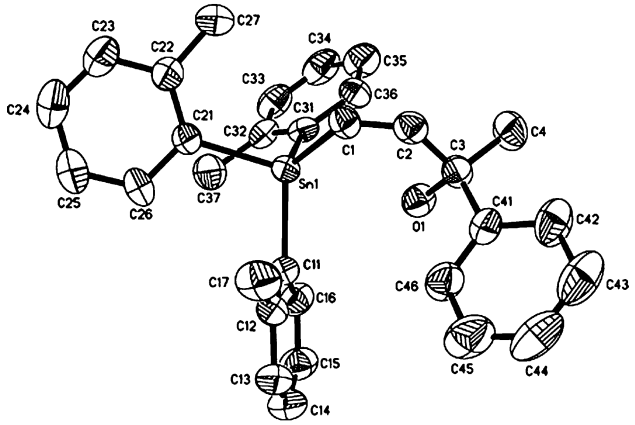


Fig. 1. The molecular structure and crystallographic numbering scheme for compound **1**.

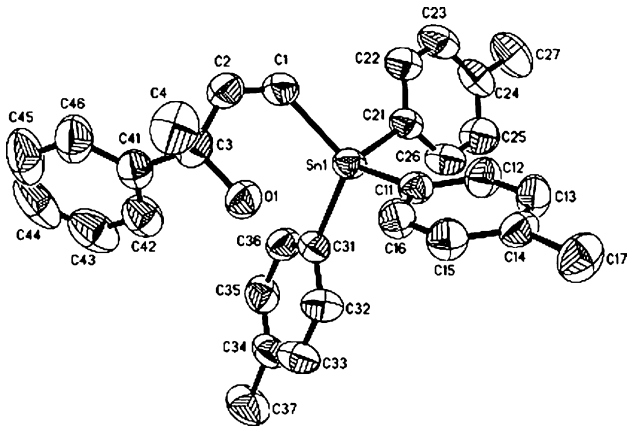


Fig. 2. The molecular structure and crystallographic numbering scheme for compound **2**.

vinyl residue, adopts a distorted tetrahedral geometry with C–Sn–C angles ranging from 103.37(19)° to 115.78(19)°. The C(1)–Sn(1)–C(11) angle (115.78(19)°) 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 Sn(1) atoms is 2.879(5) Å, which is significantly less than the sum of their van der Waals radii [3.70 Å] [18]). The weak coordination of the O(1) atom also influences the strength of the Sn(1)–C(*o*-tolyl) bond. As a result, Sn(1)–C(21) is longer by 0.03 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 reaction [2] might be attributed to the weak intramolecular O → Sn coordination. The geometry about the Sn atom in **2** is essentially the same as that for **1** with the C–Sn–C angles ranging from 102.91(14)° to 116.67(14)°.

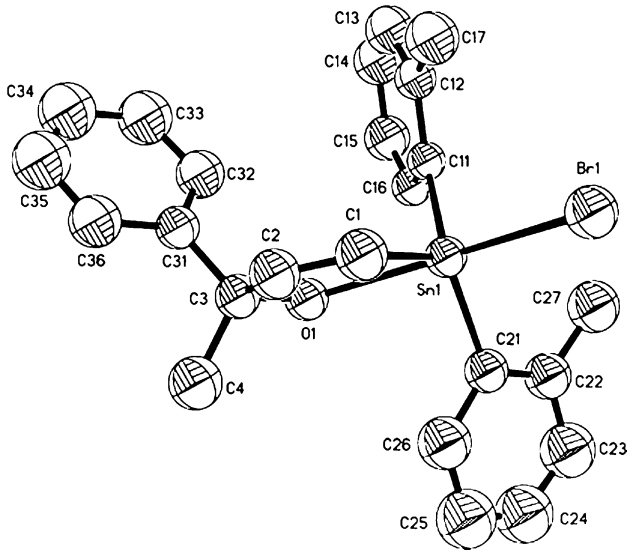


Fig. 3. The molecular structure and crystallographic numbering scheme for compound **5**.

The C(1)–Sn(1)–C(31) angle of 116.67(14)° is much wider than the corresponding angle in **1** due to stronger interaction between the O(1) atom and the Sn atom in **2**, which can be clearly seen from the relatively short Sn···O(1) distance (2.859(4) Å). These results also reflect greater Lewis acidity of the Sn atom in **2** than in **1**.

The Sn atom in **5** 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 Br(1) and O(1) atoms, which is similar to a reported analogue (*Z*)-1-[2-(chlorodi-*p*-tolylstannyl)vinyl]-1-cycloheptanol [11]. The Sn···O(1) distance of 2.483(4) Å is in the range of a normal Sn–O coordination bond length [2,8,11], indicating that the Sn atom in **5** is formally coordinated by the O(1) atom of the hydroxyl group and the Lewis acidity of the Sn atom in the $(o\text{-tolyl})_2SnBr$ moiety is greater than in the $(o\text{-tolyl})_3Sn$ moiety. On the other hand, the O(1)–C(3) bond (1.444(6) Å) in **5** is weaker than the corresponding bond (1.441(6) Å) in **1** due to the strong coordination interaction between the O(1) and Sn atoms. The Sn(1)–C(1)–C(2) angle (120.3(4)°) in **5** is considerably smaller than the equivalent angle (129.2(4)°) in **1** and the C(1)–C(2) bond (1.341(8) Å) in **5** is longer than the corresponding one (1.332(8) Å) in **1**, which is apparently the result of the strain from the five-membered ring.

4. Conclusions

We have synthesized and characterized (*Z*)-1-(triarylstannyl)-3-phenyl-1-buten-3-ol (aryl = *o*-tolyl, *p*-tolyl) and several monohalide derivatives. (*Z*)-1-(triarylstannyl)-3-phenyl-1-buten-3-ol adopts a distorted tetrahedral geometry, while the Sn atom in the monohalide derivatives is located inside a trigonal bipyramid.

5. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 210842 for compound **1**, CCDC No. 210843 for compound **2**, CCDC No. 210844 for compound **5**. 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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References

[1] A.J. Crowe, P.J. Smith, G. Atassi, Chem. Biol. Interact. 32 (1980) 171.
[2] H. Pan, R. Willem, J. Meunier-Piret, M. Gielen, Organometallics 9 (1990) 2199.
[3] F. Kayser, M. Biesemans, H. Pan, M. Gielen, R. Willem, Magn. Reson. Chem. 30 (1992) 877.
[4] M. Gielen, H. Pan, R. Willem, D. De Vos, B.V. Pharmachemie, Eur. Pat. 90202936.2-, 06/11/90, Antitumor compositions and compounds, Chem. Abstr., 116 (1992) 235873q.
[5] M. Gielen, P. Lelieveld, D. De Vos, H. Pan, R. Willem, M. Biesemans, H.H. Fiebig, Inorg. Chim. Acta 196 (1992) 115.
[6] F.F. Xin, S.X. Lin, F.Y. Jie, H. Pan, B. Mahieu, M. Gielen, F. Kayser, R. Willem, Z. Anorg. Allg. Chem. 620 (1994) 1006.
[7] F. Kayser, M. Biesemans, H. Pan, M. Gielen, R. Willem, J. Chem. Soc., Perkin Trans. 11 (1994) 297.
[8] F. Kayser, M. Biesemans, A. Delmotte, I. Verbruggen, I. De Borger, M. Gielen, R. Willem, Organometallics 13 (1994) 4026.
[9] R. Willem, A. Delmotte, I. De Borger, M. Biesemans, M. Gielen, F. Kayser, J. Organomet. Chem. 480 (1994) 255.
[10] F. Kayser, M. Biesemans, F. Fu, H. Pan, M. Gielen, R. Willem, J. Organomet. Chem. 486 (1994) 263.
[11] F. Fu, H. Li, D. Zhu, Q. Fang, H. Pan, E.R.T. Tiekink, F. Kayser, M. Biesemans, I. Verbruggen, R. Willem, M. Gielen, J. Organomet. Chem. 490 (1995) 163.
[12] H.C. Dai, Q.H. Ying, X.H. Wang, S.M. Yue, H.D. Pan, X. Chen, Polyhedron 17 (1998) 2503.
[13] H.D. Vercruijsse, W. De Graaf, L. Brandsma, Synth. Commun. 18 (1988) 131.
[14] H.D. Kuivila, O.F. Beumel Jr., J. Am. Chem. Soc. 83 (1961) 1246.
[15] D.H. Lorenz, P. Shapiro, A. Stern, E.I. Becker, J. Org. Chem. 28 (1964) 2335.
[16] G.M. Sheldrick, SHELX97 and SHELX97, University of Gőttingen, Gőttingen, Germany, 1997.
[17] R.M. Silverstein, C.G. Bassler, T.C. Morrill, Spectrometric Identification of Organic Compounds, 4th ed., Wiley, New York, 1981, p. 235.
[18] A. Bondi, J. Phys. Chem. 68 (1964) 441.