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Synthesis and reactions of arylselenomethylstannyl compounds, R_3SnCH_2SeAr. Crystal and molecular structure of [(*m*-methoxyphenylseleno)methyl]triphenylstannane

Philip J. Cox
School of Pharmacy, Robert Gordon's Institute of Technology, Schoolhill, Aberdeen AB9 1FR, Scotland (U.K.)

Solange M. S. V. Doidge-Harrison, Ian W. Nowell
School of Applied Sciences, Robert Gordon's Institute of Technology, St. Andrews Street, Aberdeen AB1 1HG, Scotland (U.K.)

R. Alan Howie, Andrew P. Randall and **James L. Wardell***
Department of Chemistry, University of Aberdeen, Meston Walk, Old Aberdeen AB9 2UE, Scotland (U.K.)

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Abstract

The crystal and molecular structure of [(*m*-methoxyphenylseleno)methyl]triphenylstannane, $Ph_3SnCH_2SeC_6H_4OMe$-*m* (II), has been determined. Compound II contains a slightly distorted tetrahedral tin atom with an intramolecular Sn---Se distance of 3.319(2) Å. Reactions of II and $Cy_3SnCH_2SeC_6H_4Cl$-*p* (III) (Cy = cyclohexyl) with various reagents have been studied; III reacts with $Pb(OAc)_4$, *N*-bromosuccinimide (NBS), I_2 or CF_3CO_2H to give $XCH_2SeC_6H_4Cl$-*p* (X = AcO, Br, I or H). Compound II reacts at the Ph–Sn bond with $Cl_2Pt(COD)$ (COD = cycloocta-1,5-diene), at the Sn–$CH_2(SeC_6H_4OMe$-*m*) bond with NBS, and at both bonds with I_2. Absorption maxima for charge-transfer complexes with $(NO_2C=C(CN)_2$ were recorded.

Introduction

Organostannanes containing oxygen and sulfur functional groups have been variously studied [1]. In contrast, the corresponding selenium containing compounds have attracted scant attention. This is exemplified by α-RX-alkylstannanes (I; $RXCR^1R^2SnR^2_3$: X = O, S or Se): the O and S analogues have been extensively studied [1–5], while data on I, X = Se, have been limited to preparations, spectra and a few reactions [2, 6].

We now present the crystal and molecular structure of [(*m*-methoxyphenylseleno)methyl]triphenylstannane (II) and on some reactions of II and [(*p*-chlorophenylseleno)methyl]tricyclohexylstannane (III) with tetracyanoethylene and with other reagents.

Results and discussion

The selenomethylstannyl compounds, $Ph_3SnCH_2SeC_6H_4OMe$-*m* (II) and $Cy_3SnCH_2SeC_6H_4Cl$-*p* (III), were prepared from the appropriate (iodomethyl)triorganostannane and areneselenol in the presence of a base by a similar method to that used previously [2].

Crystal and molecular structure of II

The atomic arrangement is shown in Fig. 1 and crystal packing is indicated in Fig. 2. Figure 1 shows

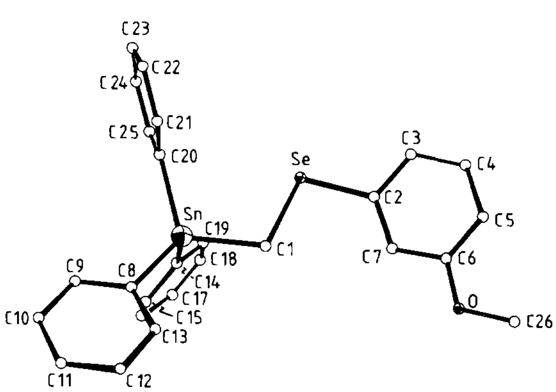

Fig. 1. X-ray molecular structure of compound II.

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*Author to whom correspondence should be addressed.

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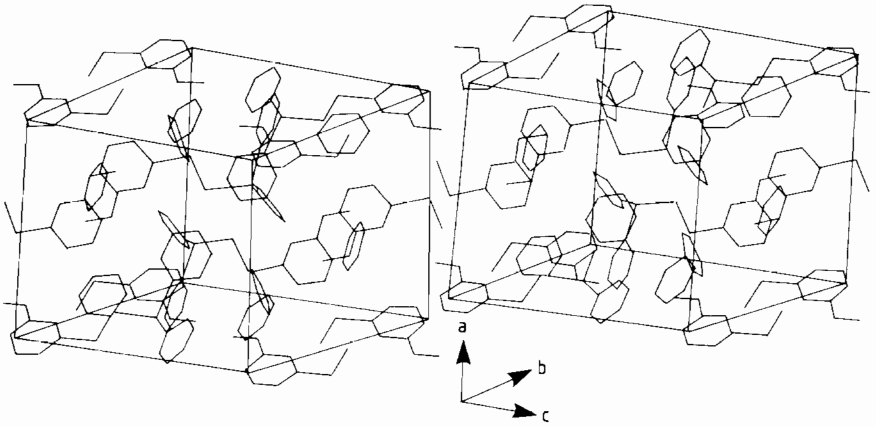

Fig. 2. Crystal structure of compound **II**.

a slightly distorted tetrahedral geometry about tin. Atomic coordinates are listed in Table 1, bond lengths in Table 2, bond angles in Table 3 and mean plane calculations in Table 4. There are no intermolecular contacts <4.0 Å.

TABLE 1. Fractional atomic coordinates ($\times 10^4$) for **II** with e.s.d.s in parentheses

	x/a	y/b	z/c	U_{eq}[a]
Sn	0.70731(4)	−0.03176(4)	0.43906(3)	0.047
O	1.0209(5)	−0.1222(5)	0.1009(4)	0.086
C(1)	0.8359(6)	−0.0537(6)	0.3733(6)	0.064
C(2)	0.9895(5)	0.0611(6)	0.2858(5)	0.052
C(3)	1.0665(6)	0.1385(7)	0.2870(6)	0.067
C(4)	1.1289(7)	0.1270(9)	0.2241(8)	0.083
C(5)	1.1173(7)	0.0415(9)	0.1620(7)	0.080
C(6)	1.0410(6)	−0.0355(8)	0.1613(6)	0.063
C(7)	0.9769(6)	−0.0252(7)	0.2230(5)	0.056
C(8)	0.6934(6)	−0.1761(6)	0.5186(5)	0.047
C(9)	0.6329(7)	−0.1781(8)	0.5828(6)	0.073
C(10)	0.6210(7)	−0.2739(9)	0.6312(7)	0.085
C(11)	0.6710(8)	−0.3680(8)	0.6153(6)	0.075
C(12)	0.7329(8)	−0.3664(7)	0.5530(6)	0.075
C(13)	0.7451(7)	−0.2724(7)	0.5054(6)	0.064
C(14)	0.5734(6)	−0.0156(6)	0.3286(5)	0.054
C(15)	0.4933(6)	−0.0910(7)	0.3232(6)	0.062
C(16)	0.4076(7)	−0.0831(9)	0.2505(8)	0.086
C(17)	0.4033(8)	0.0006(11)	0.1850(7)	0.089
C(18)	0.4810(7)	0.0737(9)	0.1915(6)	0.075
C(19)	0.5658(7)	0.0663(7)	0.2632(5)	0.060
C(20)	0.7250(5)	0.1171(6)	0.5199(5)	0.051
C(21)	0.8035(6)	0.1275(7)	0.5983(5)	0.059
C(22)	0.8157(7)	0.2244(7)	0.6492(6)	0.067
C(23)	0.7503(7)	0.3103(6)	0.6227(6)	0.064
C(24)	0.6722(7)	0.3008(7)	0.5450(6)	0.068
C(25)	0.6597(6)	0.2051(6)	0.4942(5)	0.057
C(26)	1.0861(8)	−0.1387(9)	0.0373(7)	0.093
Se	0.90307(7)	0.08573(7)	0.37177(6)	0.067

[a] $U_{eq} = \frac{1}{3}\Sigma_i\Sigma_j a^*_i a^*_j \mathbf{a}_i \cdot \mathbf{a}_j$.

TABLE 2. Bond lengths (Å) for **II** with e.s.d.s in parentheses

Sn–C(1)	2.168(9)	Sn–C(8)	2.135(7)
Sn–C(14)	2.152(8)	Sn–C(20)	2.145(8)
O–C(6)	1.36(2)	O–C(26)	1.42(2)
C(1)–Se	1.918(8)	C(2)–C(3)	1.39(2)
C(2)–C(7)	1.38(2)	C(2)–Se	1.913(8)
C(3)–C(4)	1.38(2)	C(4)–C(5)	1.37(2)
C(5)–C(6)	1.38(2)	C(6)–C(7)	1.38(2)
C(8)–C(9)	1.37(2)	C(8)–C(13)	1.39(2)
C(9)–C(10)	1.39(2)	C(10)–C(11)	1.37(2)
C(11)–C(12)	1.36(2)	C(12)–C(13)	1.36(2)
C(14)–C(15)	1.40(2)	C(14)–C(19)	1.37(2)
C(15)–C(16)	1.39(2)	C(16)–C(17)	1.39(2)
C(17)–C(18)	1.36(2)	C(18)–C(19)	1.37(2)
C(20)–C(21)	1.39(2)	C(20)–C(25)	1.38(2)
C(21)–C(22)	1.38(2)	C(22)–C(23)	1.36(2)
C(23)–C(24)	1.38(2)	C(24)–C(25)	1.37(2)

The Ph–Sn bond lengths range from 2.135(7) to 2.152(8) Å and the CH_2–Sn bond length is 2.168(9) Å. The bond angles about tin vary between 106.8(4) and 113.5(3)°. The intramolecular Sn....Se distance is 3.319(2) Å, which is well within the sum of the van der Waal's radii of Sn and Se (4.20 Å) but considerably greater than the sum of the covalent radii (2.57 Å). Comparative data for Sn–Se interactions are available from two Sn–Se bonded compounds, viz. $(PhSe)_4Sn$ (**IV**) and $Me_2SnSeSnMe_2SnMe_2Se$ (**V**). The Sn–Se bond lengths in two polymorphs [7] of **IV** were found to be in the range 2.488(2) to 2.513(2) Å. In **V**, the Sn–Se covalent bond lengths [8] were between 2.516(2) and 2.583(2) Å; in addition, intermolecular and intramolecular Sn---Se distances of 3.76–3.98 and 4.214(2)–4.242(2) Å, respectively were identified.

The C_{aryl}–Se–C_{alkyl} bond angle in **II** is 102.9(4)° and the C_{aryl}–Se and CH_2–Se bond lengths are 1.913(8) and 1.918(8) Å, respectively. Average values

TABLE 3. Valency angles (°) for **II** with e.s.d.s in parentheses

C(1)–Sn–C(8)	108.4(3)	C(1)–Sn–C(14)	106.8(4)
C(1)–Sn–C(20)	110.3(3)	C(8)–Sn–C(14)	109.2(3)
C(8)–Sn–C(20)	113.5(3)	C(14)–Sn–C(20)	108.4(3)
C(6)–O–C(26)	118.2(8)	Sn–C(1)–Se	108.5(4)
C(3)–C(2)–C(7)	120.1(8)	C(3)–C(2)–Se	116.1(6)
C(7)–C(2)–Se	123.8(6)	C(2)–C(3)–C(4)	118.6(9)
C(3)–C(4)–C(5)	121.7(9)	C(4)–C(5)–C(6)	119.6(9)
O–C(6)–C(5)	124.5(8)	O–C(6)–C(7)	115.7(8)
C(5)–C(6)–C(7)	119.8(9)	C(2)–C(7)–C(6)	120.2(8)
Sn–C(8)–C(9)	121.8(6)	Sn–C(8)–C(13)	120.4(6)
C(9)–C(8)–C(13)	117.8(9)	C(8)–C(9)–C(10)	121.2(9)
C(9)–C(10)–C(11)	119.7(9)	C(10)–C(11)–C(12)	119.7(9)
C(11)–C(12)–C(13)	120.9(9)	C(8)–C(13)–C(12)	120.7(8)
Sn–C(14)–C(15)	119.2(6)	Sn–C(14)–C(19)	121.2(6)
C(15)–C(14)–C(19)	119.7(8)	C(14)–C(15)–C(16)	119.6(9)
C(15)–C(16)–C(17)	119.0(10)	C(16)–C(17)–C(18)	120.8(10)
C(17)–C(18)–C(19)	120.4(10)	C(14)–C(19)–C(18)	120.5(9)
Sn–C(20)–C(21)	120.6(6)	Sn–C(20)–C(25)	120.8(6)
C(21)–C(20)–C(25)	118.6(7)	C(20)–C(21)–C(22)	120.3(8)
C(21)–C(22)–C(23)	120.2(8)	C(22)–C(23)–C(24)	120.0(8)
C(23)–C(24)–C(25)	120.2(8)	C(20)–C(25)–C(24)	120.7(8)
C(1)–Se–C(2)	102.9(4)		

TABLE 4. Mean-plane calculations[a] (Å) for **II**

(1) *Sn* 0.0289(5), *C*(1) 0.489(8), Se −0.0189(9), C(2) 0.019(8), C(3) 0.014(9), C(4) −0.007(10), C(5) −0.011(10), C(6) −0.002(9), C(7) 0.007(8)

[a] Italicized atoms not included in derivation of mean plane.

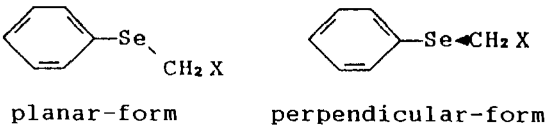

Fig. 3. Conformations of $PhSeCH_2X$.

of bond lengths taken from a number of crystal structure determinations [9] of organic selenides are 1.93 Å for C_{aryl}–Se and 1.97 Å for C_{alkyl}–Se.

Normal values for C–Se–C bond angles for C_{aryl}–Sn and C_{alkyl}–Se bond lengths would result in a Sn---Se distance of 3.2–3.4 Å. The small angular values for C(1)–Sn...Se (33°) and the values for the bond angles about tin do not give any indication of a Sn---Se interaction. Of interest, the Sn atom is barely out (by 0.03 Å) of the plane containing the Se atom and the C_6H_4OMe-*m* ring; on the other hand, the α-methylene carbon (C_1) lies well outside this plane (by 0.49 Å).

From a gas-phase, electron-diffraction study of $PhSeCH_3$, the predominant conformer was calculated to have the methyl carbon out of the plane containing the Se atom and the phenyl ring [10], other features being C_{alkyl}–Se and C_{aryl}–Se bond lengths of 1.957(19) and 1.912(2) Å, respectively with a C–Se–C bond angle of 99.6(25)°. However, two rotamers—a planar ($IP = 8.00$ eV) and a perpendicular form ($IP = 8.30$ eV) in a 1:1 ratio—were indicated [11] from a photoelectron spectral study of $PhSeCH_3$ (see Fig. 3). The planar form is the one in which the overlap of the Se(4p) and π-system is at a maximum. Increasing steric hinderance by X in $ArSeCH_2X$ leads to decreasing proportions of the planar form. Compound **II** in the solid state clearly exists as the out-of-plane conformer.

Interactions with $(NC)_2C{=}C(CN)_2$ and iodine

Arenes [12], including aryl sulphides [13] and selenides [14], form charge-transfer complexes with π-acceptors, such as $(NC)_2C{=}C(CN)_2$. From the values for the maximum absorptions (λ_{max}) of complexes with $(NC)_2C{=}C(CN)_2$, values for the ionization potentials may be calculated using the empirical relationship, eqn. (1) [12].

$$(\lambda_{max})^{-1}\ (\mathrm{cm}^{-1}) = 7333Ip - 41830 \qquad (1)$$

Values for the ionization potentials for **II** and **III**, calculated from λ_{max} values for $(NC)_2C{=}C(CN)_2$ complexes in CH_2Cl_2 solution, are given in Table 5, along with data for alkyl aryl selenides and for some sulfur analogues.The ionization potential refer to MOs mainly localized at the Se(S)-ring fragment.[*]

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[*] A referee pointed out that the similarities of the ionization potential values suggest similar C_1–S(Se)–C_2–C_7 dihedral angles. The value for this in solid **II** is 17.40 while in solid $Cy_3SnCH_2SC_6H_4Cl$-*p* the value is 9.7° [16].

TABLE 5. Charge transfer absorption maxima for complexes between $(NC)_2C{=}C(CN)_2$ and alkyl aryl selenides and sulfides in CH_2Cl_2 solution

Compound	λ_{max} (nm)	Ip[a] (eV)	Compound	λ_{max} (nm)	Ip[a] (eV)
MeSePh	601[b]	7.99[b], 8.00[c]	MeSPh	572[d]	8.10[b], 8.07[e]
$MeSeC_6H_4Cl$-*p*	603[b]	7.95[b]	$MeSC_6H_4Cl$-*p*	572[d]	8.09[b], 8.07[e]
$Cy_3SnCH_2SeC_6H_4Cl$-*p*	641[f]	7.83[f]	$Cy_3SnCH_2SC_6H_4Cl$-*p*	621[g]	7.90[g]
$Ph_3SnCH_2SeC_6H_4OMe$-*m*	626[f]	7.88[f]	$Ph_3SnCH_2SC_6H_4Cl$-*p*	598[d]	7.99[d]

[a] Ionisation potentials, either directly measured or calculated from $\lambda\ cm^{-1} = 7331Ip - 41830$. [b] Ref. 14. [c] Ref. 11. [d] Ref. 3. [e] Ref. 15. [f] This study. [g] Ref. 16.

The $(NC)_2C{=}C(CN)_2$ complexes of **II** and **III**—unlike those of the sulfur analogues—are only stable for short times.

Iodine (a σ-acceptor) can also form complexes with organic selenides [17]. Complex formation between I_2 and **II** or **III** is indicated by the generation of intense yellow–orange coloration; fairly rapid reactions ensue. An approximate value for λ_{max} (355 nm) was found for the I_2: **III** complex in CH_2Cl_2 solution, values for $ArSeCH_3$ complexes in non-polar solution occur [17] between 330 and 350 nm.

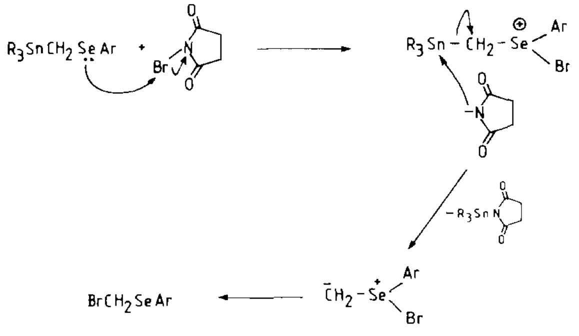
The products of the reaction of **II** with I_2 occur by cleavage of both carbon–tin bonds, eqn. (2)

$$\mathbf{II} + I_2 \begin{array}{l} \nearrow Ph_3SnI + ICH_2SeC_6H_4OMe\text{-}m \\ \searrow IPh_2SnCH_2SeC_6H_4OMe\text{-}m + PhI \end{array} \qquad (2)$$

Products of reaction between **III** and I_2 are Cy_3SnI and $ICH_2SeC_6H_4Cl$-*p* (**VI**) (eqn. 3)). Compound (**VI**) (δCH_2 4.23) slowly converts to $CH_2(SeC_6H_4Cl\text{-}p)_2$ (δCH_2 4.10) in the reaction solution.

Other reactions

N-Bromosuccinimide (NBS) reacted [18] with **II** to give mainly $BrCH_2SeC_6H_4OMe$-*m* and $Ph_3SnNCOCH_2CH_2CO$. Normally NBS would be expected to cleave an aryl–tin bond of an alkylaryltin compound such as **II**. In the reaction with **II**, it is envisaged that NBS reacted at the Se atom initially with the subsequent reaction as shown in Scheme 1.



Scheme 1.

A similar mechanism was proposed for the reaction of NBS with $Cy_3SnCH_2SC_6H_4Cl$-*p* [16] and of course with $Cy_3SnCH_2SeC_6H_4Cl$-*p* (this study). The compound $Cl_2Pt(COD)$ (COD = cycloocta-1,5-diene), cleaved Ph–Sn bonds in **II** to produce initially $Cl_nPh_{3-n}SnCH_2Se_6H_4OMe$-*m* (**VII**; $n = 1$) and $Cl_{2-n}Ph_nPt(COD)$ (**VIII**, $n = 1$) and subsequently (**VII**; $n = 2$) and (**VIII**; $n = 2$).

Cyclohexyl–tin bonds are much less reactive than are phenyl–tin bonds; any reaction of **III** which occurred was at the $Sn{-}CH_2$ bond. Thus, NBS, $Pb(OAc)_4$ or CF_3CO_2H reacted with **III** at room temperature (r.t.) to give as the major selenium containing product, $XCH_2SeC_6H_4Cl$-*p* (X = Br, AcO or H, respectively) (eqns. (3) and (4)) (see Table 6).

$$\mathbf{III} + Y\text{-}X \longrightarrow Cy_3SnY + XCH_2SeC_6H_4Cl\text{-}p \qquad (3)$$

Y-X = $COCH_2CH_2CON$-Br, CF_3CO_2-H, I-I

$$\mathbf{III} + Pb(OAc)_4 \xrightarrow{-Cy_3SnOAc} [(AcO)_3PbCH_2SeC_6H_4Cl\text{-}p] \longrightarrow AcOCH_2SeC_6H_4Cl\text{-}p + Pb(OAc)_2 \qquad (4)$$

The $Pb(OAc)_4$ reaction probably proceeds via the exchange product, $(AcO)_3PbCH_2SeC_6H_4Cl$-*p*, which quickly collapses to $Pb(OAc)_2$ and $AcOCH_2SeC_6H_4Cl$-*p*. Alkyl–lead triacetates are reported to be thermally labile and unisolatable [19]. Pinhey and co-workers [20] have previously used $Pb(OAc)_4$/organostannane exchanges to generate the more stable aryl–, heteroaryl– and vinyl–lead triacetate compounds.

Neither $Cl_2Pt(COD)$ nor $Hg(OAc)_2$ reacted with **III** at r.t. even after weeks.

Experimental

Melting points (m.p.) were measured on a Kofler hotstage and are uncorrected. ^1H NMR spectra were obtained on a Perkin-Elmer R34 (220 MHz) spectrometer and ^{119}Sn NMR spectra obtained on a Jeol FX90 instrument; $\delta^{119}Sn$ relative to Me_4Sn. UV–Vis spectra were recorded on a Perkin-Elmer Lambda 15 UV–Vis spectrophotometer.

Compounds, Ph_3SnCH_2I and Cy_3SnCH_2I, were obtained from the appropriate chlorotriorganotin, CH_2I_2 and a zinc/copper couple by a published procedure [21].

The (arylselenomethyl)triorganotin compounds were obtained from the reaction of the appropriate (iodomethyl)triorganotin and areneselenol in EtOH in the presence of base [2].

$Ph_3SnCH_2SeC_6H_4OMe$-*m*, m.p. 51–52 °C (EtOH). *Anal.* Found: C, 56.6; H, 4.5. Calc. for $C_{26}H_{24}OSeSn$: C, 56.8; H, 4.3%. δH ($CDCl_3$; 220 MHz): 2.74 (2H, s, $J^{119}Sn–^1H$ 43 Hz, CH_2), 3.50 (3H, s, OMe), [6,68 (1H, dd), 6.95 (1H, d), 7.00 (1H, d), 7.12 (1H, t) C_6H_4], [7.38 (9H, m, *meta*+*para*), 7.58 (6H, m, *ortho*) Ph_3Sn].

$Cy_3SnCH_2SeC_6H_4Cl$-*p*, m.p. 76–77 °C (needles, EtOH). *Anal.* Found: C, 52.3; H, 6.9; Cl, 6.2. Calc. for $C_{25}H_{39}ClSeSn$: C, 52.4; H, 6.9; Cl, 6.2%. δH (CCl_4: 220 MHz); 1.2–2.0 (33H, m, Cy); 2.07 (2H, s, $J^{119}Sn–^1H$ 36 Hz, CH_2), [7.14 (2H, d, J 7 Hz) and 7.28 (2H, d, J 7 Hz), C_6H_4]. $\delta^{119}Sn$ ($CDCl_3$) −62.5 ppm.

Compound $Cl_2Pt(COD)$ was obtained by a published procedure [22]. All other reagents were purified commercial samples.

Reactions of II and III

Equimolar solutions of II or III and the reagent were mixed at room temperature. The progress of the reactions were monitored by ^1H NMR spectroscopy. ^{119}Sn NMR spectroscopy was used to identify the organotin product. Data are given in Table 6.

Crystal structure determination of compound II

Crystal data

$C_{26}H_{24}OSeSn$, $M = 550.11$, monoclinic, space group $P2_1/c$, $a = 13.404(15)$, $b = 12.125(9)$, $c = 14.658(17)$ Å,

TABLE 6. Products of equimolar reaction (0.15–0.25 M) between **II** or **III** and reagents at room temperature

Compound	Reagent/Solvent	Selenium containing products	Other products[a]
II	NBS/CD_2Cl_2	$BrCH_2SeC_6H_4OMe$-*m* [δCH_2: 4.74][b]	unknown [δH 5.90] $Ph_3Sn\overline{NCOCH_2CH_2CO}$ [δCH_2 2.61]
	I_2/CCl_4	$ICH_2SeC_6H_4OMe$-*m*[c] [δCH_2: 4.23][d]	PhI Ph_3SnI [$\delta^{119}Sn$ −114]
	$Cl_2Pt(COD)/CD_2Cl_2$	$Ph_2ClSnCH_2SeC_6H_4OMe$-*m* [δCH_2 2.96] $PhCl_2SnCH_2SeC_6H_4OMe$-*m* [δCH_2 3.21] both unstable	PhClPt(COD) [δH 5.70 (t, 37Hz); 4.55 (t, 77.5Hz) olefinic] $Ph_2Pt(COD)$ [δH 5.02 (t, 40Hz) olefinic]
III	NBS/CD_2Cl_2	$BrCH_2SeC_6H_4Cl$-*p* [δCH_2: 4.74]	unknown [δH 5.89] $Cy_3Sn\overline{NCOCH_2CH_2CO}$ [δH 1.2–2.0 (Cy) 2.60(CH_2)]; $\delta^{119}Sn$ +79.6
	I_2/CCl_4	$ICH_2SeC_6H_4Cl$-*p* [δH: 4.21 (2H, s, CH_2), 7.27 (2H, d) and 7.46 (2H, d) aryl][d]	Cy_3SnI[$\delta^{119}Sn$ +65.6]
	CF_3CO_2H/CCl_4[e]	$CH_3SeC_6H_4Cl$-*p* [δH 2.27 (3H, s, Me), 7.16 (2H, d) and 7.31 (2s, d) aryl][f]	$Cy_3SnO_2CCF_3$ [$\delta^{119}Sn$ +7.0]
	$Pb(OAc)_4/CDCl_3$	$CH_3CO_2CH_2SeC_6H_4Cl$-*p* [δH 2.05 (3H, s, CH_3), 5.52 (2H, s, CH_2) 7.26 (2H, d) and 7.63 (2H, d) aryl]	$Pb(OAc)_2$ [δH 2.10 (CH_3)] unknown [δH 3.31] Cy_3SnOAc [$\delta^{119}Sn$ +9.0]
	$Cl_2Pt(COD)/CD_2Cl_2$/7d	no reaction	
	$Hg(OAc)_2/CD_2Cl_2$/7d[g]	no reaction	

[a] $\delta^{119}Sn$ relative to Me_4Sn. [b] δCH_2 of $PhSeCH_2Br(CCl_4)$: 4.65 [23]: δCH_2 *p*-$MeC_6H_4SeCH_2Br(CCl_4)$ 4.73 [2]. [c] $IPh_2SnCH_2SeC_6H_4OMe$-*m* also formed but decomposed quickly. [d] δCH_2 of *p*-$MeC_6H_4SeCH_2I(CCl_4)$: 4.23 [2]. [e] Five-fold excess taken. [f] δCH_2 of CH_3SeAr 2.27 [24]; δCH_3 of $CH_3SePh(CCl_4)$: 2.30 [25]. [g] Suspension.

$\beta = 102.52(8)°$, $V = 2330(3)$ Å, $Z = 4$, $D_c = 1.57$ g cm^{-3}, $D_m = 1.61$ g cm^{-3}, $F(000) = 1087.95$, μ(Mo Kα) = 19.6 cm^{-1}, $\lambda = 0.71069$ Å, T = room temperature.

Data collection and processing

Colourless crystal, $0.60 \times 0.24 \times 0.34$ mm. The cell dimensions were obtained from setting angles of 14 independent reflections with $2\theta \sim 21°$ on a Nicolet P3 automated diffractometer using monochromated Mo Kα radiation. A total of 6829 unique intensities ($0 < \theta < 30°$) were measured by the $\omega/2\theta$ scan technique; 3688 reflections had $F > 5\sigma(F)$. Range of hkl: $0 < h < 20$, $0 < k < 18$, $-21 < l < 21$. The data were corrected for Lorentz and polarization effects but absorption was ignored. Two reference reflections, monitored periodically, showed no significant variation in intensity.

Structure analysis and refinement

The structure was determined by the heavy-atom method (Patterson function) which revealed the approximate position of the tin atom. The remaining non-hydrogen atoms were located [26] from successive Fourier difference maps using SHELX 76. All hydrogen atoms were located but given ideal geometry. Full matrix least-squares calculations on F with anisotropic thermal parameters for the tin, selenium, oxygen and carbons, and isotropic thermal parameters for hydrogens converged at R 0.0515 and R_w 0.0515. Atomic scattering factors were from SHELX 76 and the International Tables for X-ray Crystallography [27].

Final $w = 1.5239/\sigma^2$ (F_o), Δ/σ 0.004, final $\Delta\rho_{min} = -0.77$ e Å^{-3}, final $\Delta\rho_{max} = 1.08$ e Å^{-3}.

Molecular geometries were generated by the GX package [28].

Supplementary material

Lists of anisotropic thermal parameters, H-atom positions, and tables of F_o and F_c are available from the Cambridge Crystallographic Data Centre.

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