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TRIORGANOTIN ARYL SELENIDES

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Summary

The Sn–Se bond in trimethyltin aryl selenides is cleaved on reaction with selenenyl halides, sulphenyl halides, alkyl halides and allyl halides. Thus $Me_3SnSePh$ reacts with PhSeCl, 4-Me-2-$NO_2C_6H_3SCl$, RI ($R = CH_3$ or Ph_3SnCH_2) and $CH_2{=}CHCH_2Br$ to give PhSeSePh, 4-Me-2-$NO_2C_6H_3SSePh$, RSePh and $CH_2{=}CHCH_2SePh$, respectively. The diselenide, PhSeSePh, is also obtained from $Me_3SnSePh$ on reaction with either 4-$MeC_6H_4SO_2Cl$ or $NaIO_4$. Exchange reactions also occur between $Me_3SnSePh$ and Ph_3SnCl or PhHgCl.

Introduction

Triorganotin sulphides, R_3SnSR', have been frequently studied, and many reactions reported [1]. In contrast, the selenide analogues, R_3SnSeR', have only attracted little attention, as indicated in a recent survey [2]. Relatively few reactions of R_3SnSeR have been studied; among them are those with I_2 [3], Ac_2O [4] and HBr [5]. In all these cases cleavage of the Sn–Se bond occurred:

$$R_3SnSeR' \xrightarrow{I_2} R_3SnI + R'SeSeR' \qquad (1)$$

$$R_3SnSeR' \xrightarrow{Ac_2O} R_3SnOAc + AcSeR' \qquad (2)$$

$$R_3SnSeR' \xrightarrow{HBr} R_3SnBr + HSeR' \qquad (3)$$

In this paper, we wish to report some further reactions and preparations of R_3SnSeR' compounds.

Experimental

Areneselenols were gifts from Dr. W. MacFarlane; additional compounds were obtained as required by reaction of Se with aryl-Grignard reagents [6].

Preparation of triorganotin aryl selenides. All preparations were carried out under nitrogen. Equimolar quantities of R_3SnCl and ArSeH were dissolved in CCl_4 and excess Et_3N added. After stirring for 30 minutes at room temperature, the precipitate of Et_3NHCl was filtered off and the filtrate evaporated. The residues were fractionally distilled in vacuo (for trimethyltin derivatives) or recrystallised from ethanol (for the triphenyltin compound).

Trimethyltin phenyl selenide, $Me_3SnSePh$, b.p. 72°C/0.05 mmHg [lit. [7], 67–69°C/0.001 mmHg). Anal. Found: C, 34.0; H, 4.3; $C_9H_{14}SeSn$ calcd.: C, 33.7; H, 4.4%. ^{1}H NMR (60 MHz in CCl_4); δ 0.40 (s, 9H, Me_3Sn, $J(^{117,119}Sn-^1H)$ 56, 59 Hz); 7.15 (m, 3H, *m*- and *p*-protons of Ph), 7.50 ppm (m, 2H, *o*-protons of Ph).

Trimethyltin p-tolyl selenide, $Me_3SnSeC_6H_4Me$-*p*, b.p. 84–85°C/0.2 mmHg. Anal. Found: C, 36.1; H, 4.7. $C_{10}H_{16}SeSn$ calcd.: C, 36.0; H, 4.8%. ^{1}H NMR (60 MHz in CCl_4): δ 0.38 (s, 9H, Me_3Sn, $J(^{117,119}Sn-^1H)$ 54, 56 Hz); 2.33 (s, 3H, Me), 6.84 (d) and 7.27 (d) ppm (4H, C_6H_4, AB system, *J* 8 Hz).

Trimethyltin m-tolyl selenide, $Me_3SnSeC_6H_4Me$-*m*, b.p. 78–79°C/0.1 mmHg. Anal. Found: C, 36.0; H, 4.7. $C_{10}H_{16}SeSn$ calcd.: C, 36.0; H, 4.8%. ^{1}H NMR (60 MHz in CCl_4): δ 0.39 (s, 9H, Me_3Sn, $J(^{117,119}Sn-^1H)$ 55, 57 Hz), 2.39 (s, 3H, Me), 6.4–7.4 ppm (m, 4H, C_6H_4).

Trimethyltin p-anisyl selenide, $Me_3SnSeC_6H_4OMe$-*p*, b.p. 105–106°C/0.05 mmHg. Anal. Found: C, 34.1; H, 4.8. $C_{10}H_{16}OSeSn$ calcd.: C, 34.3; H, 4.6%. ^{1}H NMR (60 MHz in CCl_4): δ 0.37 (s, 3H, Me_3Sn), $J(^{117,119}Sn-^1H)$ 56, 59 Hz), 3.70 (s, 3H, MeO), 6.60 (d) and 7.30 (d) ppm (4H, C_6H_4, AB system, *J* 8 Hz).

Trimethyltin m-anisyl selenide, $Me_3SnSeC_6H_4OMe$-*m*, b.p. 110–111°C/0.05 mmHg. Anal. Found: C, 34.6; H, 4.4. $C_{10}H_{16}OSeSn$ calcd.: C, 34.3; H, 4.6%. ^{1}H NMR (60 MHz in CCl_4): δ 0.41 (s, 9H, Me_3Sn, $J(^{117,119}Sn-^1H)$ 54 and 56 Hz), 3.74 (s, 3, MeO), 6.6–7.2 (m, 4H, C_6H_4).

Trimethyltin m-trifluoromethylphenyl selenide, $Me_3SnSeC_6H_4CF_3$-*m*, b.p. 70–71°C/0.05 mmHg. Anal. Found: C, 30.9; H, 3.3. $C_{10}H_{13}F_3SeSn$ calcd.: C, 31.0; H, 3.4%. ^{1}H NMR (60 MHz in CCl_4): δ 0.44 (s, 9H, Me_3Sn, $J(^{117,119}Sn-^1H)$ 56, 59 Hz), 7.2–7.8 ppm (m, 4H, C_6H_4).

Triphenyltin p-tolyl selenide, $Ph_3SnSeC_6H_4Me$-*p*, m.p. 92°C. Anal. Found: C, 57.8; H, 4.1. $C_{25}H_{22}SeSn$ calcd.: C, 57.8; H, 4.3%. ^{1}H NMR (60 MHz in CCl_4): δ 2.20 (s, 3H, Me), 6.75 (d, 2H, A portion of C_6H_4 AB system, *J* 8 Hz), 7.0–7.7 ppm (m, 17H, Ph_3Sn + B portion of C_6H_4 AB system).

Reagents. Methyl iodide was shaken with dilute KOH solution, washed well with H_2O, dried over $CaCl_2$ and redistilled. 4-Methyl-2-nitrobenzenesulphenyl chloride was prepared as published [8]. Phenylmercury chloride, m.p. 253–256°C [9] and Ph_3SnCH_2I, m.p. 85–86.5°C [10] were from previous studies. Other reagents were commercial samples.

Reactions

Trimethyltin phenyl selenide and iodine. A solution of iodine (0.508 g, 2 mmol) in CCl_4 was added dropwise to a solution of $Me_3SnSePh$ (0.636 g, 2 mmol). Reaction was rapid and produced Me_3SnI (δ 0.86, $J(^{117,119}Sn-^1H)$, 54, 57 Hz) and PhSeSePh [δ 7.22 (m, 4H, *o*-protons), 7.58 ppm (m, 6H, *m*- and *p*-protons)]. Diphenyl diselenide, m.p. 61–62°C (lit. [3] 61°C) was obtained from the reaction mixture by TLC on silica gel.

Triphenyltin phenyl selenide and phenylselenenyl chloride. A solution of PhSeCl

(0.076 g, 0.4 mmol) in $CDCl_3$ (0.5 ml) was dropped into a solution of $Me_3SnSePh$ (0.128 g, 0.4 mmol) in CCl_4 (0.5 ml). Reaction was immediate with the formation of Me_3SnCl (^1H NMR δ 0.64 ppm) and PhSeSePh; the latter was obtained by TlC on silica gel. It had m.p. 61–63 °C.

Trimethyltin phenyl selenide and 4-methyl-2-nitrobenzenesulphenyl chloride. Solutions of $Me_3SnSePh$ (0.636 g, 2 mmol) and $4\text{-Me-2-}NO_2C_6H_3SCl$ (0.407 g, 2 mmol) in CCl_4 were mixed and the reaction mixture stirred. Reaction was complete after 1 h; ^1H NMR spectrum indicated the complete formation of Me_3SnCl (δ 0.64, ppm $J(^{117,119}Sn-^1H)$ 54, 57 Hz). Work-up of the reaction mixture by TLC led to isolation of $4\text{-Me-2-}NO_2C_6H_3SSePh$ (0.41 g, 63%), m.p. 84–87 °C. (^1H NMR 100 MHz in $CDCl_3$, δ 2.38 (s, 3H, Me), 7.2–8.0 ppm (m, 8H, aryl). Anal. Found: C, 48.3; H, 3.6; N, 4.2; S, 10.1. $C_{13}H_{11}NO_2SSe$ calcd.: C, 48.4; H, 3.4; N, 4.3; S, 9.9%) and small quantities of PhSeSePh, m.p. 61 °C (^1H NMR 100 MHz in $CDCl_3$, δ 7.22 (m, 4H, *o*-protons), 7.58 ppm (m, 6H, *m*- and *p*-protons)) and $(4\text{-Me-2-}NO_2C_6H_3S)_2$ (^1H NMR 100 MHz in $CDCl_3$; δ 2.40 (s, 6H, Me), 7.4–8.1 ppm (m, 6H, aryl)).

Trimethyltin phenyl selenide and methyl iodide. Trimethyltin phenyl selenide (43 mg, 0.135 mmol) was dissolved in methyl iodide (0.3 ml). Formation of Me_3SnI was followed by ^1H NMR spectroscopy over a period of 24 h. Considerable broadening of the Me_3Sn absorptions were noted during the course of the exchange reaction. Evaporation of excess MeI, at the end of the reaction, under reduced pressure at room temperature left a residue containing Me_3SnI (^1H NMR in $CDCl_3$ δ 0.87) and MeSePh (^1H NMR in $CDCl_3$: δ 2.34 (s, 3H, Me), 7.1–7.5 ppm (m, 5H, aryl)). The latter was collected as an oil using TLC on silica gel.

Slower reactions occurred between MeI and $Me_3SnSePh$ (1/1 and 2/1 mol ratios) in $CDCl_3$ solution at room temperature.

Trimethyltin phenyl selenide and iodomethyltriphenyltin. A solution of $Me_3SnSePh$ (0.159 g, 0.5 mmol) and Ph_3SnCH_2I (0.246 g, 0.5 mmol) in $CDCl_3$ (1 ml) was maintained at 34 °C. The reaction was monitored by ^1H NMR; the slow formation (50% reaction after 7 d) of Ph_3SnCH_2SePh ($\delta(CH_2)$ 2.87 ppm, $J(^{117,119}Sn-^1H)$ 42, 44 Hz) was indicated. A single broad Me_3Sn signal was observed during the exchange reaction. Removal of most of the solvent and addition of hexane to the residue resulted in the precipitation of Ph_3SnCH_2SePh, 0.18 g, m.p. 100–102 °C. Anal. Found: C, 57.9; H, 4.4. $C_{25}H_{22}SeSn$ calcd.: C, 57.7; H, 4.3%.

Trimethyltin phenyl selenide and allyl bromide. A slow reaction occurred between allyl bromide (0.053 g, 0.44 mmol) and $Me_3SnSePh$ (0.139 g, 0.44 mmol) in $CDCl_3$ (1 ml) at 34 °C the formation of allyl selenide was indicated from the ^1H NMR spectrum by comparison with that reported for $CH_2{=}CHCH_2SePh$ [11]. Allyl phenyl sulphide was collected as an oil from the reaction mixture using TLC on silica gel.

Trimethyltin phenyl selenide and triphenyltin chloride. A solution of $Me_3SnSePh$ (32 mg, 0.1 mmol) and Ph_3SnCl (38.5 mg, 0.1 mmol) was prepared in $CDCl_3$. Complete exchange occurred as shown by the presence in the ^1H NMR spectrum of absorption of Me_3SnCl (δ 0.65 ppm) and absence of absorptions of $Me_3SnSePh$ (δ 0.40 ppm).

The ^1H NMR spectrum of a solution of $Me_3SnSePh$ and Ph_3SnCl (mol. ratio 2/1) indicated the presence of equivalent quantities of Me_3SnCl and $Me_3SnSePh$. Both Me_3Sn signals remained sharp and separate.

Trimethyltin phenyl selenide and phenylmercury chloride. A solution of PhHgCl

(31.2 mg, 0.1 mmol) and $Me_3SnSePh$ (32 mg, 0.1 mmol) was made up in $CDCl_3$. ^1H NMR spectroscopy indicated the complete formation of Me_3SnCl (δ 0.65 ppm) and complete consumption of $Me_3SnSePh$. On work-up, the initial PhHgSePh product symmetrised to Ph_2Hg and $(PhSe)_2Hg$.

Trimethyltin phenyl selenide and sodium periodate. A solution of $Me_3SnSePh$ (0.428 g, 1.34 mmol) in dioxane (10 ml) was added to a solution of $NaIO_4$ (0.286 g) in H_2O. After leaving overnight, the white solid was filtered off and the filtrate evaporated under reduced pressure at room temperature. The yellow-coloured residue was chromatographed on thin layers: a yellow product, PhSeSePh, 0.150 g, 71%, m.p. 61–62 °C was collected.

Trimethyltin phenyl selenide and p-toluenesulphonyl chloride. (a) A solution of $Me_3SnSePh$ (0.114 g, 0.36 mmol) and p-$MeC_6H_4SO_2Cl$ (0.068 g, 0.36 mmol) was made up in $CDCl_3$ (1 ml). The ^1H NMR spectrum, after 24 h, reaction, showed a single Me_3Sn absorption (δ 0.58, $J(^{117,119}Sn-^1H)$ 59, 62 Hz) and two major Me absorptions (δ 2.24 and 2.30 ppm). Further slow reactions occurred on standing at 34 °C. TLC of the reaction mixture (using silica gel with 60–80 °C pet. ether as eluant) provided PhSeSePh, yield 0.070 g.

(b) A solution of $Me_3SnSePh$ (0.114 g, 0.36 mmol) and p-$MeC_6H_4SO_2Cl$ (0.034 0.18 mmol) was made up in $CDCl_3$ (1 ml). The ^1H NMR spectrum, after 3 days, showed a single Me_3Sn absorption (δ 0.70 ppm, $J(^{117,119}-^1H)$ 63,66 Hz) and one major Me absorption (δ 2.48 ppm) little further change occurred on standing. TLC of the reaction mixture (on silica gel) using pet-ether as eluant provided PhSeSePh m.p. 61 °C (yield 0.051 g).

Results and discussion

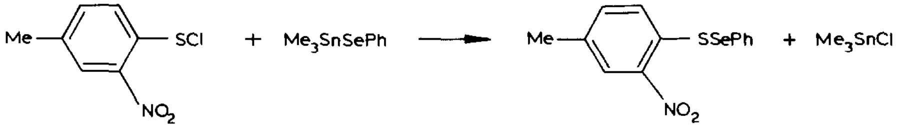
Previously used methods of preparation of triorganotin selenides, R_3SnSeR', have included reactions of (i) R_3SnX (X = Cl or Br) and either HSeR′ (in the presence of a base [3]), R′SeNa [3,12] or $(RSe)_4Al$ [5], (ii) R_3SnOR'' [4,13] or $(R_3Sn)_2O$ [3] with R′SeH, (iii) $R_3SnNR''_2$ and R′SeH [5,13], (iv) R_3SnNCO and R′SeH [14], (v) R_3SnH and R′SePh [15] and (vi) Ph_4Sn and Se [16]. The first of these methods ($Me_3SnCl/R'SeH/NEt_3$) was used in this study to produce a number of $Me_3SnSeC_6H_4X$ compounds (X = H, m-Me, p-Me, m-MeO, p-MeO and m-CF_3). As previously reported for R_3SnSeR' compounds, all the $Me_3SnSeC_6H_4X$ compounds possessed significant stability towards air and water. Some darkening and decomposition of samples, kept in capped bottles, was however noted after several months, the products being $(XC_6H_4Se)_2$ and $(Me_3Sn)_2O$. Treatment of $Me_3SnSePh$ with the oxidant $NaIO_4$ in aqueous dioxane rapidly gave the diselenide, PhSeSePh.

Reactions of a number of electrophiles were studied with $Me_3SnSePh$. Cleavage of the Sn–Se bond was found to occur on reaction with I_2, selenenyl halides, sulphenyl halides, sulphonyl halides, alkyl iodides or allyl halides, in addition exchange reactions occurred with Ph_3SnCl or PhHgCl.

As shown also by MacMullin and Peach [3] reaction with I_2 leads to the symmetric diselenide, eq. 1. It seemed probable that the course of the reaction proceeded via PhSeI. To confirm that selenenyl halides can cleave Sn–Se bonds, the reaction of PhSeCl was studied with $Me_3SnSePh$ eq. 4. ^1H NMR spectroscopy indicated that immediate and complete reaction occurred at room temperature,

$$PhSeCl + Me_3SnSePh \rightarrow Me_3SnCl + PhSeSePh \qquad (4)$$

although work-up, using TLC, only led to an isolated yield of PhSeSePh of 70%. The compound, PhSeCl, also reacted quantitatively with other $Me_3SnSeC_6H_4X$ compounds in NMR scale reactions (ca. 0.06 mmol) to give Me_3SnCl and the unsymmetrical diselenides, $PhSeSeC_6H_4X$, contaminated with much smaller amounts of the two diselenides, PhSeSePh and $XC_6H_4SeSeC_6H_4X$. These latter products probably arise from symmetrization of $PhSeSeC_6H_4X$, a process previously noted for unsymmetrical diselenides, [6].



(5)

Reaction of the sulphenyl halide, $4\text{-Me-2-NO}_2C_6H_3SCl$, with $Me_3SnSePh$ produced the seleneyl sulphide, eq. 5. This product was particularly readily symmetrised to PhSeSePh and $(4\text{-Me-2-NO}_2C_6H_3S)_2$, for example on heating or on standing in solution; however it could be isolated in good yield with care from the reaction mixture. There have been several reports of the ready symmetrization of RSeSR′ species [6]. The R_3SnSeR' route to selenenyl sulphides has synthetic potential and is a useful addition to those routes already known [6].

A more complex reaction occurs between $p\text{-MeC}_6H_4SO_2Cl$ and $Me_3SnSePh$. Both 1/1 and 1/2 reactions ($p\text{-MeC}_6H_4SO_2Cl/Me_3SnSePh$) were studied; in each case, the shift in the Me_3Sn signal in the ^1H NMR spectra in $CDCl_3$ solution (from δ 0.40 to 0.65 ppm) signified reaction. Two major species (approximately 1/1) containing $p\text{-MeC}_6H_4SO_2$ fragments (δ(Me) 2.25 and 2.30 ppm) were present in the solution after 30 h at 34°C when a 1/1 mole ratio of reagents were used; a 1/2 ratio of reagents, however, provided essentially one $p\text{-MeC}_6H_4SO_2$ containing product (δ(Me) 2.35 ppm) *. These findings appear analogous to those observed for $Ph_4SbSAr/p\text{-MeC}_6H_4SO_2Cl$ reactions, in which the initial $p\text{-MeC}_6H_4SO_2SAr$ product was also reactive [17]; hence eq. 6 and 7 are suggested

$$Me_3SnSePh + p\text{-MeC}_6H_4SO_2Cl \rightarrow Me_3SnCl + PhSeSO_2C_6H_4Me\text{-}p \qquad (6)$$

$$Me_3SnSePh + PhSeSO_2C_6H_4Me\text{-}p \rightarrow Me_3SnOS(O)C_6H_4\text{-}p + PhSeSePh \qquad (7)$$

TLC of the products from both the 1/1 and 1/2 reactions led to the isolation of only PhSeSePh. Fong and Kitching have reported the ^1H NMR spectrum of $Me_3SnOS(O)C_6H_4Me\text{-}p$ in $CDCl_3$ solution [18] ($\delta(Me_3Sn)$ 0.55 ppm ($J(^{119}Sn-^1H)$ 70 Hz), δ(Me) 2.40 ppm). The Me_3Sn chemical shift value is a little different from those observed in the ^1H NMR spectra of the $p\text{-MeC}_6H_4SO_2Cl/Me_3SnSePh$ reaction mixtures; this difference could arise from the presence of other Me_3Sn species (e.g. Me_3SnCl) in the reaction mixtures which give rise to average $\delta(Me_3Sn)$ values as a consequence of rapid exchange reactions.

The selenosulphonate, $PhSeSO_2C_6H_4Me\text{-}p$ is known; It is a reactive molecule; for example, it decomposes readily in light at room temperature or on heating and it adds readily to alkenes, by a free radical mechanism [19].

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* Further slow reactions occurred in the 1/1 reaction on standing; this included formation of a dimethyltin (IV) compound (δ 1.20 ppm) and of $p\text{-MeC}_6H_4SO_2Me$ (δ(Me) 2.92 ppm).

Alkyl iodides (MeI and Ph_3SnCH_2I) and $CH_2{=}CHCH_2Br$ react slowly with $Me_3SnSePh$ at 34°C in chloroform solution, eq. 8 and 9. During the course of each of these reactions only a broad single Me_3Sn signal was observed in the ^1H NMR spectrum, indicating a rapid exchange, on the NMR time scale, between Me_3SnX (X = Br or I) and $Me_3SnSePh$. The reaction between MeI and $Me_3SnSePh$ contrasts with the lack of reaction between MeI and $Bu_3SnSePh$, as reported by MacMullin and Peach [3].

$$Me_3SnSePh + RCH_2I \rightarrow Me_3SnI + RCH_2SePh \tag{8}$$

(R = H or Ph_3Sn)

$$Me_3SnSePh + CH_2{=}CHCH_2Br \rightarrow Me_3SnBr + CH_2{=}CHCH_2SePh \tag{9}$$

Complete anion exchange occurs between $Me_3SnSePh$ and Ph_3SnCl or PhHgCl, eq. 10 and 11. The organomercury product of eq. 11 readily undergoes symmetrization and could not be isolated. In these exchange reactions involving chlorides, two distinct Me_3Sn signals were observed during the exchanges.

$$Me_3SnSePh + Ph_3SnCl \rightarrow Me_3SnCl + Ph_3SnSePh \tag{10}$$

$$Me_3SnSePh + PhHgCl \rightarrow Me_3SnCl + PhHgSePh \tag{11}$$

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