Organometallic Chemistry

Oxidation of γ-stannyl sulfides by monochloramine CB and hydrogen peroxide

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The first γ-trimethylstannyl sulfimide, $Me_3Sn(CH_2)_3S(=NSO_2Ar)C_5H_{11}$-*n*, was synthesized by oxidative imination of $Me_3Sn(CH_2)_3SC_5H_{11}$-*n* with $ArSO_2N(Na)Cl$ (Ar = C_6H_4Cl-4). Oxidation of γ-trimethylstannyl sulfimide by an alkaline solution of H_2O_2 gave γ-trimethylstannyl sulfoximide, $Me_3Sn(CH_2)_3S(O)(=NSO_2Ar)C_5H_{11}$-*n*, and γ-trimethylstannyl sulfone, $Me_3Sn(CH_2)_3SO_2C_5H_{11}$-*n*, the latter compound resulting from hydrolysis of the arylsulfimide group. Oxidation of stannyl sulfide by hydrogen peroxide yielded γ-trimethylstannyl sulfoxide, $Me_3Sn(CH_2)_3S(O)C_5H_{11}$-*n* (under mild conditions) or γ-trimethylstannyl sulfone (under more severe conditions).

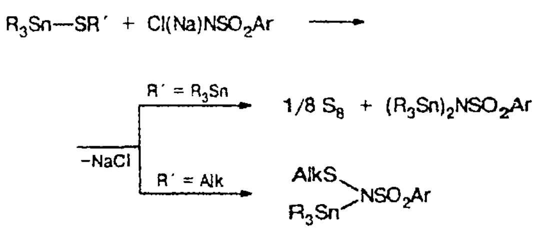
Key words: pentyl 3-(trimetylstannyl)propyl sulfide, oxidation, γ-trimethylstannyl sulfimide, γ-trimethylstannyl sulfoximide, γ-trimethylstannyl sulfoxide, γ-trimethylstannyl sulfone.

Previously,[1,2] we have demonstrated that sulfides RSR′ (R = SnR_3; and R′ = SnR_3 or Alk) readily react with such compounds as chloramines, for example, with $ArSO_2N(Na)Cl$, where Ar = C_6H_4Cl-4 (monochloramine CB).

It is known[3–7] that analogous reactions of sulfides RSR′ (R and R′ = Alk or Ar) afford products of the oxidative imination, namely, sulfimides $RS(=NSO_2Ar)R'$, which are also called iminosulfuranes,[4] sulfylimines,[3,5,6] sulfinimines,[7] or sulfinylimines.[7] In this work, we use the name "sulfimide" accepted in the monograph (see Ref. 4).

Unlike organic sulfides, in organotin sulfides containing more polar metal—sulfur bonds, these bonds are always cleaved (desulfurization). Thus, trialkyltin sulfides $(Alk_3Sn)_2S$ undergo oxidative desulfurization under the action of monochloramine-CB to produce elemental sulfur in quantitative yield.[1] In the case of trialkyltin thioalkoxides, desulfurization is accompanied by the transfer of the alkylthio group to the nitrogen atom of the sulfamide fragment[2] (Scheme 1).

Scheme 1



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From these results it follows that oxidative imination cannot be carried out with sulfides in which the sulfur atom is directly bonded to the trialkylstannyl group. However, tin-containing sulfides $R_3Sn(CH_2)_nSR'$, in which tin and sulfur atoms are separated by a polymethylene bridge, would be expected to be oxidized by monochloramine CB to form tin-containing sulfimides $R_3Sn(CH_2)_nS(=NSO_2C_5H_4Cl\text{-}4)R'$. The analogs of these compounds of general formula $R_2R'Sn(CH_2)_nS(O)_mR''$ are known[8—14]: tin-containing sulfoxides {m = 1: R = R' = Bu, n = 1, and R" = Me, or Ph [8]; R = R' = *cyclo*-Hex, n = 1, and R" = 4-ClC_6H_4 (characterized by the ^{119}Sn NMR spectra in solution[9]); R = R' = Ph, n = 3, or 4, and R" = 4-MeC_6H_4 [10]; or R = Ph, R' = Cl, Br, or I, n = 2, 3, or 4, and R" = 4-MeC_6H_4 [10,11]} and sulfones {m = 2: R = R' = Bu, n = 1, and R" = Bu, 4-ClC_6H_4, or 4-MeOC_6H_4 [12], Ph [12,13]; R = R' = Ph, n = 2, 3, or 4, and R" = 4-MeC_6H_4 [10]; and R = Ph, R' = Cl, Br, or I, n = 2, 3, or 4, and R" = 4-MeC_6H_4 [10,11]}. Some of these compounds exhibit pesticide activity.[8,14]

The above-mentioned tin-containing sulfoxides and sulfones were obtained by the exchange reaction of a halide atom or an amino group at the tin atom for the sulfenyl- or sulfonyl-alkyl group,[8,12,13] by hydrostannylation of sulfones containing a terminal double bond,[10,14] and by direct oxidation of tin-containing sulfides by 4-chloroperoxybenzoic acid.[9,10]

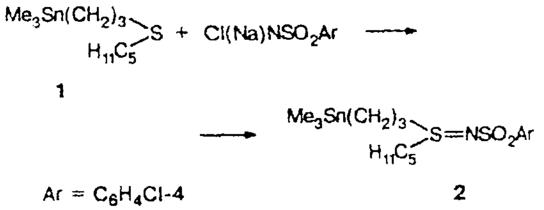
As part of our continuing studies on oxidation of organotin sulfur-containing compounds,[1,2,15,16] in this work we carried out the reaction of γ-trialkylstannyl sulfide with chloramine-CB and hydrogen peroxide.

Results and Discussion

We studied oxidation of tin-containing sulfides using $Me_3Sn(CH_2)_3SC_5H_{11}\text{-}n$ (**1**), which was obtained by the UV-induced addition of pentanethiol to allyltrimethyltin at −70 °C, as an example.

Oxidation of sulfide **1** by chloramine CB in MeOH occurs at ~20 °C with elimination of NaCl. When the reaction mixture was heated over a short period, the yield of NaCl was almost quantitative. As expected, the product of the imination, *S*-pentyl-*S*-[3-(trimethylstannyl)propyl]-*N*-(4-chlorophenyl) sulfimide (**2**), was the major identified organotin compound (Scheme 2).

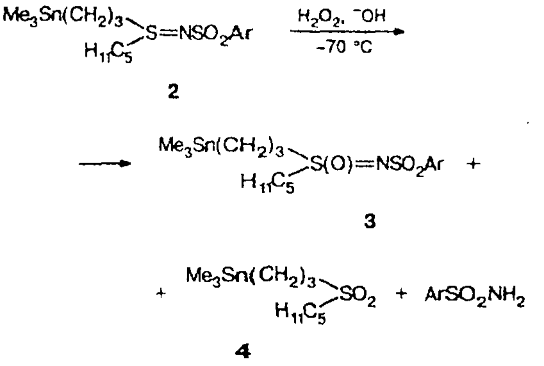
Scheme 2



Sulfur is tetravalent in sulfimides. Under the action of strong oxidizers (potassium permanganate and alkaline hydrogen peroxide), sulfur can be oxidized further to form sulfoximides.[3,5,17]

Actually, sulfimide **2** oxidizes by alkaline hydrogen peroxide in boiling MeOH with retention of the trimethylstannyl group. An oxidation product, *S*-pentyl-*S*-[3-(trimethylstannyl)propyl]-*N*-(4-chlorophenylsulfonyl) sulfoximide (**3**), was isolated from the reaction mixture in a yield of ~21%. Pentyl 3-(trimethylstannyl)propyl sulfone (**4**), which formed as a result of alkaline hydrolysis of the sulfimide group in compound **3**, was the major tin-containing product (the yield was ~67%). As expected, the ratio of sulfone **4** and 4-chlorobenzenesulfamide is virtually equimolar. Taking into account the ratio between organotin products **3** and **4**, on the one hand, the ratio between product **4** and sulfamide, on the other hand, and a virtually complete conversion of the initial sulfimide **2**, it can be assumed that most of sulfoximide **3**, which is initially formed, transforms into sulfone **4** under reaction conditions. The overall process can be described by Scheme 3.

Scheme 3



Additional experiments demonstrated that a decrease in the reaction time decreases the conversion of the initial sulfide, whereas an increase in the reaction time favors the hydrolysis of sulfoximide **3** and increases the yield of sulfone **4**.

Oxidation of sulfide **1** by hydrogen peroxide can be used as an alternative procedure for the preparation of sulfone **4**. Under mild conditions (acetone, 0—20 °C),[18] the reaction is stopped at the stage of formation of pentyl 3-(trimethylstannyl)propyl sulfoxide (**5**), whereas under more severe conditions (MeOH, NaOH, ~70 °C), more complete oxidation of **1** occurs to form sulfone **4**.

The structures of new compounds **1—5** were confirmed by the data of elemental analysis and ^{1}H NMR and IR spectroscopy.

The IR spectrum of sulfimide **2** shows absorption band of the arylsulfimide group, namely, the bands of the SO_2 group (1300 and 1140 cm^{-1}), the $N—S^{VI}$ bond (930 cm^{-1}),[19] and the newly formed $N{=}S^{IV}$ bond (1075 and 995 cm^{-1}).[4] The IR spectrum of sulfoxide **5** contains only the band of the $S^{IV}{=}O$ sulfoxide group with the maximum at 1025 cm^{-1}.

The IR spectrum of sulfoximide **3** contains the absorption band of the $S^{VI}{=}O$ bond at 1200 cm^{-1}, which shifts to the short-wavelength region by 175 cm^{-1} compared to that observed for sulfoxide **5**, apparently due to the effect of the electron-withdrawing arylsulfimide group. The $S^{VI}{=}O$ group affects the positions of some characteristic bands in the spectrum of compound **3**. It is known[5] that the S=N sulfimide bond in compounds of type **2** is highly polarizable, and the position of the absorption band of this bond substantially depends on the character of additional substituents at the sulfur atom. Because of this, in the spectrum of sulfoximide **3**, the maximum of one of two bands, which we attribute to this bond, substantially shifts to the short-wave region: 1060 cm^{-1} (995 cm^{-1} in the spectrum of sulfimide **2**), which agrees with the published data.[17] The position of the second band at 1080 cm^{-1} remains virtually unchanged (1075 cm^{-1} in the spectrum of sulfimide **2**).

The IR spectrum of sulfone **4** is much simpler that those of compounds **2** and **3**. The absorption bands of the aromatic ring (1580 cm^{-1}), the $S^{VI}{=}O$ group (1200 cm^{-1}), the $N{=}S^{IV}$ (1075 and 995 cm^{-1}) or $N{=}S^{VI}$ sulfimide bond (1080 and 1060 cm^{-1}), and the $N—S^{VI}$ sulfamide bond (910 cm^{-1}) disappear, but intense characteristic bands of the SO_2 group with the maxima at 1270 and 1135 cm^{-1} are observed.

In the ^1H NMR spectra of compounds **1—5**, the assignment of the signals of all protons was made. When sulfide **1** oxidizes to sulfone **5**, the electronegative SO_2 group appears, which leads to the downfield shift of the signals of β-protons and, particularly, α-protons, which agrees with the published data.[20] An analogous (but less pronounced) effect was observed also in the case of sulfoximide **3** compared to sulfimide **2**, apparently due to the additional oxidation of the sulfur atom in **2**. In the NMR spectra of all compounds, satellites were observed around the signals of the trimethylstannyl groups, which occur through spin-spin coupling of protons with magnetic isotopes of tin. In all cases, spin-spin coupling constants are in the range of 53.0—53.5 ($^2J_{^{119}Sn—H}$) and 51.5—52.0 Hz ($^2J_{^{117}Sn—H}$).

Experimental

Commercial technical-grade chloramine CB was purified, and active chlorine was determined according to the known procedure.[21]

The IR spectra were recorded on a Perkin—Elmer 577 instrument (pure liquid for **1** and **5** or Nujol mulls for **2—4**).

The ^1H NMR spectra were obtained on a DC-567A instrument (100 MHz) in $CDCl_3$. Hexamethyldisiloxane was used as the internal standard.

Quantitative determination of sulfur in the presence of halide in compounds **2** and **3** was carried out by combustion according to the Schoniger procedure.[22]

Pentyl 3-(trimethylstannyl)propyl sulfide (1). A mixture of allyltrimethyltin (11.85 g, 57.9 mmol), 1-pentanethiol (9.05 g, 86.5 mmol), and benzoyl peroxide (0.1185 g) in anhydrous hexane (65 mL) was placed into a quartz vessel and irradiated with UV light (a PRK-7 lamp) for 10 h with cooling (−50— −70 °C) and stirring. The solvent and the excess pentanethiol were removed under reduced pressure first at room temperature and then at 70—75 °C. The residue was distilled *in vacuo*. Sulfide **1** was obtained in a yield of 14.5 g (81%), b.p. 121—122 °C (4 Torr); n_D^{25} 1.5004. Found (%): C, 42.41; H, 8.50; S, 10.54; Sn, 38.19. $C_{11}H_{26}SSn$. Calculated (%): C, 42.76; H, 8.42; S, 10.37; Sn, 38.45. IR, ν/cm^{-1}: 760 (ρ(Sn—Me)); 710 (C—S—C); 520 (Sn—C). ^1H NMR, δ: 0.05 (s, 9 H, Me_3Sn); 0.8—1.0 (m, 5 H, C—Me, $Sn—CH_2$); 1.2—1.5 (m, 4 H, $(\underline{CH}_2)_2—Me$); 1.4—1.9 (m, 4 H, $\underline{CH}_2—CH_2SCH_2—\underline{CH}_2$); 2.49 (t, 4 H, CH_2SCH_2, J = 7.3 Hz).

Oxidative imination of sulfide 1. A solution of monocloramine-CB (95.5% of active chlorine, 1.86 g, 7.2 mmol) in MeOH (15 mL) was added portionwise with stirring to a suspension of sulfide **1** (2.01 g, 6.5 mmol) in MeOH (10 mL) with exclusion of air. A weak exothermic effect was observed, and NaCl sedimented as a milk-white precipitate. The reaction mixture was heated to 100 °C for 3 h. Subsequent operations were carried out in air. The precipitate of NaCl was filtered off. The filtrate was concentrated, and the residue was treated with CH_2Cl_2 (30 mL). The remaining NaCl was filtered off. The total yield of NaCl was 0.41 g (98%). The filtrate was concentrated *in vacuo*. The viscous residue (3.19 g) was triturated with ether (40 mL), and a crude product was filtered off in a yield of 2.71 g. Crystallization from heptane (50 mL) produced sulfimide **2** in a yield of 2.01 g (62%), m.p. 95—96 °C. Found (%): C, 41.12; H, 6.08; S, 13.07; Sn, 23.58. $C_{17}H_{30}ClNO_2S_2Sn$. Calculated (%): C, 40.95; H, 6.02; S, 12.85; Sn, 23.83. IR, ν/cm^{-1}: 1290 ($\nu_{as}(SO_2)$); 1130 ($\nu_s(SO_2)$); 990 (S=N); 760 (ρ(Sn—Me)); 525 (Sn—C). ^1H NMR, δ: 0.05 (s, 9 H, Me_3Sn); 0.5—0.8 (m, 2 H, $Sn—CH_2$); 0.8 (m, 3 H, C—Me); 1.1—1.25 (m, 4 H, $(\underline{CH}_2)_2—Me$); 1.4—1.9 (m, 4 H, $\underline{CH}_2—CH_2SCH_2—\underline{CH}_2$); 2.5—3.0 (m, 4 H, CH_2SCH_2); 7.25—7.9 (AA´BB´, 4 H, C_6H_4).

The ethereal extract was concentrated *in vacuo*, and a viscous unidentified tin-containing residue was obtained in a yield of 1.08 g. The IR spectrum of this residue contains absorption bands observed also in the spectra of sulfimide **2** and 4-chlorobenzenesulfamide.

Oxidation of sulfimide 2 by hydrogen peroxide. A 30% solution of H_2O_2 (44 mL) and a 10% NaOH solution (5 mL) were added to a solution of sulfimide **2** (1.42 g, 2.8 mmol) in MeOH (90 mL). The reaction mixture was boiled under reflux for 2 h. Then NaOH (5 mL) was added. The reaction mixture was boiled for 5 h, cooled to room temperature, and kept for one day. The colorless crystalline compound that formed was separated by decantation, washed with cold dilute MeOH (3 mL), and dried in air. The product (0.5 g) was triturated with pentane (10 mL). The residue insoluble in pentane was recrystallized from hexane to give sulfoximide **3** in a yield of 0.31 g (21.6%), m.p. 67—68.5 °C. Found (%): C, 39.21; H, 5.89; S, 12.39; Sn, 23.13. $C_{17}H_{30}ClNO_3S_2Sn$. Calculated (%): C, 39.67; H, 5.83; S, 12.45; Sn, 23.08. IR, ν/cm^{-1}: 1290 ($\nu_{as}(SO_2)$); 1190 (S=O); 1140 ($\nu_s(SO_2)$); 1060 (S=N); 760 (ρ(Sn—Me)); 520 (Sn—C). ^1H NMR, δ: 0.10 (s, 9 H, Me_3Sn); 0.7—1.0 (m, 5 H, C—Me, $Sn—CH_2$); 1.2—1.5 (m, 4 H, $(\underline{CH}_2)_2$—Me); 1.6—2.2 (m, 4 H, $\underline{CH}_2$—CH_2SCH_2—$\underline{CH}_2$); 3.2—3.4 (m, 4 H, CH_2SCH_2); 7.38—7.92 (AA'BB', 4 H, C_6H_4).

The pentane solution was concentrated to one-half of the initial volume. Sulfone **4** was isolated by freezing at −10 °C as a colorless crystalline compound in a yield of 0.17 g, m.p. 35.5—36 °C. Found (%): C, 39.01; H, 7.90; S, 9.22; Sn, 34.80. $C_{11}H_{26}O_2SSn$. Calculated (%): C, 38.74; H, 7.63; S, 9.39; Sn, 34.84. IR, ν/cm^{-1}: 1300, 1270 ($\nu_{as}(SO_2)$); 1130—1115 ($\nu_s(SO_2)$); 760 (ρ(Sn—Me)). ^{1}H NMR, δ: 0.09 (s, 9 H, Me_3Sn); 0.8—1.0 (m, 5 H, C—Me, Sn—CH_2); 1.2—1.5 (m, 4 H, $(\underline{CH}_2)_2$—Me); 1.6—2.2 (m, 4 H, $\underline{CH}_2$—CH_2SCH_2—$\underline{CH}_2$); 2.95 (t, 4 H, CH_2SCH_2, J = 7.7, 8.0 Hz).

The aqueous-methanolic solution was extracted with CCl_4 (3×25 mL). The extract was dried with $CaCl_2$. The solvent was removed *in vacuo*. The viscous residue (0.51 g) was triturated with pentane (25 mL). The residue insoluble in pentane (0.03 g) was the initial sulfimide **2** (2.1%) (its IR spectrum was identical to that of pure compound **2**). The pentane solution was frozen at −10 °C to give sulfone **4** in a yield of 0.47 g. The total yield of sulfone **4** was 0.64 g (67.7%). The aqueous-methanolic solution that remained after extraction with CCl_4 was concentrated to ~30 mL, and 4-chlorobenzenesulfamide was extracted with ether (3×20 mL) in a yield of 0.35 g (65%), m.p. 140—142 °C.

Oxidation of sulfide 1 by hydrogen peroxide. ***A.*** A 31.6% H_2O_2 solution (1.5 g, 10.7 mmol) in acetone (5 mL) was added dropwise with stirring to a solution of **1** (3.30 g, 10.7 mmol) in acetone (20 mL) at 0 °C. The reaction mixture was kept at 0 °C overnight and then at ~20 °C for 5 h. Then the reaction mixture was boiled under reflux for 1 h. The solvent was removed *in vacuo*. The residue was distilled. The unconsumed initial sulfide was recovered in a yield of 1.0 g (30%) (the fraction with the b.p. 119—123 °C (3 Torr)), and sulfoxide **5** was obtained in a yield of 1.89 g (54%) as a pale-yellow liquid, b.p. 161—163 °C (3 Torr); n_D^{25} 1.5102. Found (%): C, 40.38; H, 8.46; S, 9.03; Sn, 36.74. $C_{11}H_{26}OSSn$. Calculated (%): C, 40.65; H, 8.01; S, 9.85; Sn, 36.56. IR, ν/cm^{-1}: 1025 (S=O); 765 (ρ(Sn—Me)); 720 (C—S—C); 525 (Sn—C). ^{1}H NMR, δ: 0.08 (s, 9 H, Me_3Sn); 0.77—1.0 (m, 5 H, C—Me, Sn—CH_2); 1.28—1.52 (m, 4 H, $(\underline{CH}_2)_2$—Me); 1.6—2.2 (m, 4 H, $\underline{CH}_2$—CH_2SCH_2—$\underline{CH}_2$); 2.56—2.78 (m, 4 H, CH_2SCH_2). The yield of a resinous unidentified residue obtained after distillation was 0.16 g.

B. A 10% NaOH solution (40 mL) and a 31.6% H_2O_2 solution (11.6 g, 82.7 mmol) were added to a solution of sulfide **1** (3.34 g, 10.8 mmol) in MeOH (50 mL). The two-phase mixture was refluxed with intense stirring for 1 h. The KI-test for peroxides was negative. Then the above-described procedure with the addition of the same amounts of H_2O_2 followed by reflux for 2—3 h was repeated five times. The reaction mixture was extracted with chloroform (4×40 mL). The extract was washed with water (50 mL) and dried with $CaCl_2$. The solvent was removed under reduced pressure, and the residue was distilled *in vacuo*. The initial sulfide **1** was obtained in a yield of 1.2 g (3.9 mmol), b.p. 85—87 °C (0.5 Torr), and a fraction that contained sulfone **4** and a small impurity of sulfoxide **5** was obtained in a yield of 1.73 g, b.p. 140—145 °C (0.5 Torr). This fraction was dissolved in hexane (50 mL) and frozen at −10 °C to produce sulfone **4** in a yield of 1.55 g (42%), m.p. 38—39 °C, whose IR spectrum was identical to that of the pure compound. The hexane filtrate was concentrated, and sulfoxide **5** was obtained in a yield of 0.18 g (5%), whose IR spectrum was identical to that of the pure compound.

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