Lithiation of the Tin-containing Sulfone Me₃SnCH₂CH₂CH₂SO₂C₆H₄CH₃-4

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Abstract — The tin-containing sulfide $Me_3Sn(CH_2)_3$ –S–C₆H₅CH₃-4 obtained by photoaddition of 4-toluenethiol to allyltrimethyltin was oxidized with hydrogen peroxide to synthesize the tin-containing sulfone $Me_3Sn(CH_2)_3$ –SO₂–C₆H₄CH₃-4, the tin and sulfur atoms in which are separated by a trimethylene bribge. Treatment of the sulfone with butyllithium gave a first tin-containing lithium salt having a red-brown color. The exchange reaction of this salt with methyl iodide resulted in formation of two new isomeric tincontaining sulfones $Me_3SnCH_2CH_2CH_2CH(CH_3)$ –SO₂–C₆H₄CH₃ and $Me_3Sn(CH_2)_3$ –SO₂–C₆H₄CH₂CH₃ identified by ¹H NMR spectroscopy. The latter result implies that the tin-containing sulfone is lithiated both by the methylene group adjacent to the sulfonyl group and by the toluene methyl group.

Sulfones RCH_2 -SO₂-R' of the aliphatic (R = R' = Alk) or aliphatic-aromatic series (R = Alk, R' = Ar) are CH acids, since their α -methylene protons are acidified by the ajacent electron-acceptor SO₂ group. Under the action of alkalis, alkali metal alkoxides and hydrides, butyllithium, or Grignard reagents they form sulforyl carbanions as salts \overline{RCH} -SO₂- \overline{RM} which are stable at room temperature [1-3]. In our previous study on carbofunctional organotin compounds [4] we synthesized the organometallic sulfone Me₃- $Sn(CH_2)_3$ - SO_2 - C_5H_{11} . In the present work we set ourselves the task to prepare first lithium salts of such sulfones that could further be used as key compounds for extending the synthetic potential of organometallic chemistry. For the example we took the tin-containing aliphatic-aromatic sulfone 4-methylphenyl 3-(trimethylstannyl)propyl sulfone Me₃Sn(CH₂)₃-SO₂- $C_6H_4CH_3-4$ (I). In this compound the tin and sulfur atoms are separated by a trimethylene bridge but equally interesting would be the related derivative with the shorter dimethylene bridge.

Compound I was prepared by oxidation of 4-methylphenyl 3-(trimethylstannyl)propyl sulfide $Me_3Sn(CH_2)_3-S-C_6H_4CH_3-4$ (II) with hydrogen peroxide. Sulfide II, as well as pentyl 2-(trimethylstannyl)ethyl sulfide $Me_3Sn(CH_2)_2-S-C_5H_{11}$ which has a dimethylene bridge between tin and sulfur, were synthesized in a similar way by photoaddition of the corresponding thiol to allyltrimethyltin or trimethylvinyltin [4]. The addition of thiols to allyltrimethyltin occurs stereoselectively to give the target product II with an admixture of the disulfide 4-MeC₆H₄SSC₆H₄. Me-4. The addition of thiols to trimethylvinyltin occurs in a more complicated fashion (by and against Markovnikov's rule), yielding a mixture two isomeric organotin sulfides pentyl 2-(trimethylstannyl)ethyl sulfide $Me_3SnCH_2C\dot{H}_2S\dot{C}_5H_{11}$ (III) and pentyl 1-(trimethylstannyl)sulfide $Me_3SnCH(CH_3)SC_5H_{11}$ (IV). We failed to separate sulfides III and IV by usual methods and idenfied them as a mixture by ¹H and ¹³C NMR spectroscopy. The ratio of the sulfides in the mixture was found to be 5:1. In addition, three by-products were isolated and identified: 1,1-bis(trimethylstannyl)ethane (Me₃Sn)₂CHCH₃ (V), 1,2-bis-(trimethylstannyl)ethane Me₃SnCH₂CH₂SnMe₃ (VI), and 1,2-bis(1-penthylthio)ethane $C_5H_{11}SCH_2CH_2S$. C_5H_{11} (VII). The isomeric organotin compounds V and VI could also not be separated and were identified as a mixture by ¹H NMR spectroscopy. The ratio of V and VI is 4:1. The composition of products III-**VII** can be explained by the scheme proposed in [5].

Sulfides are characteristically readily susceptible to oxidative imination with sodium *N*-chloroarenesulfonamides Cl(Na)NSO₂Ar (Chloramine-T, where Ar = C₆H₄Me; Chloramine-XB, where Ar = C₆H₄Cl, etc.) to form sulfonimides. Sulfonimides readily crystallize. They have well-defined melting points and are used for identification of various silfides [6]. Earlier [7] we employed this reaction for characterization of the organotin sulfide Me₃Sn(CH₂)₃SC₅H₁₁ and obtained the first representative of tin-containing sulfonimides, Me₃Sn(CH₂)₃S(=NSO₂C₆H₄Cl-4)C₅H₁₁. In the present work sulfide **II** was characterized in a similar way as *S*-(4-methylphenyl)-*S*-[3-(trimethyl-

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stannyl)propyl]-N-(4-chlorophenylsolfonyl)sulfonimide Me₃Sn(CH₂)₃S(=NSO₂C₆H₄Cl-4)C₆H₄CH₃-4 (**VIII**). By treatment with Chloramine-XB on the mixture of sulfides **III** and **IV** we expected to obtain two isomeric sulfonimides which could further be separated. But it proved that tin-containing sulfonimides in which the trialkylstannyl and sulfonyl groups are intervened by a dimethylene bridge are rather unstable and undergo ethylene elimination on usual workup.

It was found that sulfone I reacts with butyllithium in DMSO at room temperature to give lithium salt IX as an abundant brick-red precipitate. The exchange reaction of the salt with methyl iodide occurs at room temperature; decoloration is observed immediately after mixing the reactants. We found that the reaction product X is a mixture of the isomers 1-methyl 4-methylphenyl 3-(trimethylstannyl)propyl sulfone (Xa) and 4-ethylphenyl 3-(trimethylstannyl)propyl sulfone (Xb). This result implies that sulfone I is lithiated both by a methylene group of the trimethylene bridge and by the methyl group attached to the aromatic ring, resulting in formation of lithium salts **IXa** and **IXb**. The formation of isomer **IXb** can be explained by activation of the methyl protons by the SO_2 group through the conjugation system of the aromatic ring.

$$\begin{array}{c} Me_{3}SnCH_{2}CH_{2}CH_{2}-SO_{2}-C_{6}H_{4}CH_{3}-4\\ \xrightarrow{BuLi} \rightarrow Me_{3}SnCH_{2}CH_{2}CH_{-}SO_{2}-C_{6}H_{4}CH_{3}-4\\ & \downarrow\\ Li\\ \hline IXa\\ + Me_{3}SnCH_{2}CH_{2}CH_{2}-SO_{2}-C_{6}H_{4}(CH_{2}Li)-4\\ \hline IXb\\ \xrightarrow{MeI} -LiI \rightarrow Me_{3}SnCH_{2}CH_{2}CH_{-}SO_{2}-C_{6}H_{4}CH_{3}-4\\ & Me\\ \hline Xa\\ + Me_{3}SnCH_{2}CH_{2}CH_{2}-SO_{2}-C_{6}H_{4}(CH_{2}CH_{3})-4. \end{array}$$

Xb

We failed to separate isomers **Xa** and **Xb** by vacuum distillation and column chromatography on SiO_2 . Thin-layer chromatography, too, gave two poorly resolved spots. Therefore, the isomers were identified as a mixture by ¹H NMR, and their ratio was established by the integral intensities of the following signals: CH₂SO₂ (3.07 ppm) and MeCHSO₂ C₆H₄CH₂Me (2.90)ppm) multiplets, quartet (2.75 ppm), and $C_6H_4CH_3$ singlet (2.45 ppm). As follows from ¹H NMR data, the reaction mixture contains some starting sulfone I. Therefore, the signals of the CH_2SO_2 group belong both to product Vb and to sulfone **I**. Furthermore, the signals of the $C_6H_4CH_3$ group are common for product **Xa** and sulfone **I**. According to calculations, the **Xa**: **X** isomer ratio is 1.6.

EXPERIMENTAL

The IR spectra were recorded on a Perkin–Elmer-577 instrument (neat liquid or suspension in Vaseline oil; KBr). The ¹H and ¹³C NMR spectra were measured on a Bruker DPX-200 instrument, internal reference TMS.

Technical grade Chloramine-XB was purified and its analysis for active chlorine was performed as described in [8]. Dimethyl sulfoxide was dried in the following way: 30 ml of DMSO distilled over granulated NaOH [bp 81-85°C (18 mm)] was treated with 10 ml of a 0.6 N solution of butyllithium in hexane, and the resulting mixture was repeatedly distilled collecting DMSO directly into the reaction ampule [bp 61-62°C (3 mm)]. The other solvents were purified by conventional procedures. All reactions were carried out in evacuated vessels. Photoreactions were performed under irradiation with a PRK-4 lamp. Thin-layer chromatography was performed on Silufol UV-254 plates, eluent carbon tetrachlorideacetone (7:1), development in iodine vapor. Analysis for sulfur in the presence of halogen in VIII was performed by Schoeniger.

4-Methylphenyl 3-(trimethylstannyl)propyl sulfide (II). A mixture of 12.41 g of allyltrimethyltin, 9.69 g of 4-toluenethiol, and 0.124 g of benzoyl peroxide in a 70 ml of dry hexane was placed into a quartz vessel and subjected to UV irradiation for 15 h with stirring and cooling (-50 to -70° C). The solvent was removed at reduced pressure and room temperature and then evacuated at 100°C for some hours to remove excess thiol. The residue was distilled in a vacuum to obtain two fractions. The first fraction was sulfide **II**, 13.2 g (66%), bp 138–140°C (1 mm); $n_{\rm D}^{20}$ 1.5582. IR spectrum, v, cm⁻¹: 760 (SnMe), 705 (CSC), 520 (SnC). ¹H NMR spectrum (CDCl₃), δ , ppm (J, Hz): 0.04 s (9H, Me₃Sn), d [Me₃Sn, $J_{(117}^{117}$ Sn-H) 51.6, $J_{(119}^{119}$ Sn-H) 52.6], 0.89 m (2H, SnCH₂), 1.78 m (2H, CH₂CH₂CH₂), 2.30 s (3H, $C_6H_4CH_3-4$), 2.84 m (2H, CH_2S), 7.06–7.24 (AA'BB' system; 4H, C_6H_4 Me-4). ¹³C NMR spectrum (CDCl₃), δ_C , ppm (*J*, Hz): -10.4 (3C, Me₃Sn), d [Me₃Sn, ${}^{1}J({}^{117}\text{Sn-C})$ 308.8, ${}^{1}J({}^{119}\text{Sn-C})$ 323.3], 10.1 (SnCH₂), d [SnCH₂, ${}^{1}J({}^{117}\text{Sn-C})$ 334.7, ${}^{1}J({}^{119}\text{Sn-C})$ 349.8], 20.9 (C₆H₄CH₃-4), 26.6 (CH₂CH₂CH₂), 38.3 (CH₂S), 129.5 (*o*-CH, C₆H₄), 129.8 (*m*-CH, C₆H₄), 133.1 (*p*-CH, C₆H₄), 135.6 (C–S, SC₆H₄). Found, %: C 47.74; H 6.80; S 9.67; Sn 35.68. $C_{13}H_{22}SSn$. Calculated, %: C 47.46; H 6.69; S 9.74; Sn 36.11. The second fraction [150–156°C (1 mm)] was allowed to crystallize and then recrystallized from 15 ml of pentane (-10°C) to obtain 0.76 g (8% per thiol **II**) of 4-methylphenyl disulfide, mp 45–46°C (mp 46°C [9]).

Photoreaction of trimethylvinyltin with pentanethiol. A solution of 7.5 g of trimethylvinyltin, 6.15 g of 1-pentanethiol, and 0.075 g of benzoyl peroxide in 50 ml of dry hexane was placed into a quartz vessel and subjected to UV irradiation for 15 h with stirring and cooling (-50 to -70° C). The reaction mixture was filtered at room temperature to separate 0.6 g of a solid substance not melting up to 280°C, which could not be identified. The filtrate and excess thiol were evaporated at reduced pressure (1.5 mm) and room temperature. The residue was distilled in a vacuum to obtain three fractions. The first fraction $(0.61 \text{ g}; \text{ bp } 68-70^{\circ}\text{C} (1.5 \text{ mm})]$ was an inseparable mixture of 1,1-bis(trimethylstannyl)ethane (V) and 1,2-bis(trimethylstannyl)ethane (VI). IR spectrum (thin film), v, cm⁻¹: 750 (SnMe), 520 (SnC). $(Me_3Sn)_2CHCH_3$ (V). ¹H NMR spectrum (CDCl₃), δ, ppm, (J, Hz): 0.05 s [18H, 2(CH₃)₃Sn, ²*J*(H⁻¹¹⁷/¹¹⁹Sn) 49.2/51], 0.54 q (1H, SnCH, ³*J* 7.9), 1.43 d [3H, CCH₃, ³J 7.9, ³J(H^{-117/119}Sn) 73.9/77.3]. ¹³C NMR spectrum (CDCl₃), δ_{C} , ppm (J, Hz): -9.83 $[{}^{1}J(C-{}^{117/119}Sn)$ 297.8/311.6, ${}^{3}J(C-Sn)$ 8.2], -1.96 $[{}^{1}J(C - {}^{117/119}Sn) 292.5/305.9], 14.69 [{}^{2}J(C - Sn) 24.9].$ $Me_3SnCH_2CH_2SnMe_3$ (VI). ¹H NMR spectrum (CDCl₃), δ, ppm: 0.05 s [18 H, 2(CH₃)₃Sn], 0.99 s (4H, 2CH₂). ¹³C NMR spectrum (CDCl₃), $\delta_{\rm C}$, ppm (J, Hz): $-10.67 [{}^{1}J(C-{}^{117/119}Sn) 294.7/308.5, {}^{4}J(C-Sn) 4.3], 6.84 [{}^{1}J(C-{}^{117/119}Sn) 339.2/355.6, {}^{2}J(C-{}^{117/119}Sn) 39.2/355.6, {}^{2}J(C-{}^{117/119}Sn) 39.2/35.6, {}^{2}J(C-{}^{1$ 36.0/37.6]. The ¹H NMR spectra of these isomeric compounds are consistent with those reported in [10, 11]. The second fraction [7.7 g; bp 90–96°C (1.5 mm)] was repeatedly distilled to obtain 6.3 g (54%) of a mixture of silfides III and IV, bp 71-73°C (0.5 mm). IR spectrum (thin film), v, cm^{-1} : 760 (SnMe), 525 (SnC). $Me_3SnCH_2CH_2SCH_2CH_2CH_2$. CH_2CH_3 (III). ¹H NMR spectrum (CDCl₃), δ , ppm (J, Hz): 0.10 s [9H, (CH₃)₃Sn, ²J(H^{-117/119}Sn) 51.2/53.6], 0.87–0.92 m (3H, C₄H₈CH₃), 1.14 [AA'XX' system; SnCH₂CH₂S, ³J_{AX/AX'} 10.9/6.0], 1.28–1.41 m (4H, C₂H₄CH₂CH₂CH₂CH₃), 1.60–1.72 m (2H, CH₂CH₂C₃H₇), 2.67 t (2H, ³J 7.4, SCH₂C₄H₉), 2.74 $[A\tilde{A}'XX']$ system; 2H, SnCH₂CH₂S). ¹³C NMR spectrum (CDCl₃), $\delta_{\rm C}$, ppm (*J*, Hz): -10.0 [(CH₃)₃Sn), *J*(C^{-117/119}Sn) 314.0/328.7], 11.4 [(SnCH₂CH₂S) *J*(C^{-117/119}Sn) 317.3/332.0], 13.9 (C₄H₈CH₃), 22.3 (CH₂CH₂C₃H₇), 28.9 (C₃HCH₂CH₃), 30.6 (C₂H₄CH₂·

C₂H₅), 31.6 (SCH₂C₄H₉), 39.1 (SnCH₂CH₂S). Me₃SnCH(CH₃)SCH₂CH₂CH₂CH₂CH₂CH₃ (**IV**). ¹H NMR spectrum (CDCl₃), δ, ppm (*J*, Hz): 0.14 s [(CH₃)₃Sn, ²*J*(H^{-117/119}Sn) 51.5/53.7], 1.46 d [SnCH(CH₃)S, ³*J* 7.1], 2.36 q [SnCH(CH₃)S, ³*J* 7.1], the pentyl proton signals are the same as in the above spectrum. The third fraction [1.81 g, bp 122–124°C (1.5 mm)] was repeatedly distilled to obtain 1.75 g of the bis-sulfide *n*-C₅H₁₁SCH₂CH₂SC₅H₁₁-*n* (**VII**), bp 148–149°C (5 mm). ¹H NMR spectrum (CDCl₃), δ, ppm (*J*, Hz): 0.90 t (6H, CH₃, *J* 7.0), 1.31–1.40 m [8H, S(CH₂)₂· (CH₂)₂CH₃], 1.46–1.63 m (4H, SCH₂CH₂C₃H₇), 2.54 t (4H, SCH₂C₄H₉, *J* 7.3), 2.72 s (4H, SCH₂· CH₂S). The unidentified still bottoms were 0.81 g.

S-(4-Methylphenyl)-S-[3-(trimethylstannyl)propyl]-N-(4-chlorophenylsulfonyl)sulfonimide (VIII). A solution of 1.65 g of Chloramine-XB (98% by active chlorine) in 20 ml of MeOH was added with stirring to a solution of 1.95 g of sulfide II in 20 ml of MeOH. The reaction mixture was heated under reflux for 8 h, the NaCl residue was filtered off, the filtrate was evaporated to dryness, the residue was treated with 25 ml of CH₂Cl₂, and an additional NaCl was filtered off. The overall yield of NaCl was 0.38 g (100%). The filtrate was evaporated in a vacuum, and the residue was extracted with ether in a Soxhlet apparatus to obtain 2.85 g (93%) of sulfonimide VIII, mp 111–112°C. IR spectrum, v, cm⁻¹: 1290–1260, 1070 [SO₂ (as)], 1130 [SO₂ (s)], 970 (SN^{IV}), 925 (SN^{IV}) , 755 (SnMe); 520 (SnC). ¹H NMR spectrum (CDCl₃), δ , ppm (*J*, Hz): 0.024 s (6H, Me₃Sn), d [Me₃Sn, *J*(¹¹⁷Sn-H) 51.4, *J*(¹¹⁹Sn-H) 53.6], 0.677 and 0.750 m (2H, Sn-CH₂), 1.70 m (2H, CH₂CH₂CH₂), 2.40 s (3H, $C_6H_4CH_3-4$), 2.84 and 3.106 m (2H, CH₂S), 7.27–7.55 (AA'BB' system; 4H, C_6H_4 Me-4), 7.29–7.79 (AA'BB' system; 4H, C_6H_4 Cl-4). ¹³C NMR spectrum (CDCl₃), $\delta_{\rm C}$, ppm (J, Hz): -10.4 (3C, Me₃Sn), d [Me₃Sn, $J(^{117}$ Sn–C) 317.0, $J(^{119}$ Sn–C) 331.6], 8.8 (SnCH₂), d [SnCH₂, J(¹¹⁷Sn-C) 312.8, $J(^{119}\text{Sn-C})$ 326.8], 20.7 (CH₂CH₂CH₂), 21.3 (C₆H₄C · H₃), 57.6 (CH₂S), 126.1 (o-CH, C₆H₄Me-4), 127.7 (o-CH, C₆H₄Cl-4), 128.6 (m-CH, C₆H₄Cl-4), 130.5 (m-CH, C₆H₄Me-4), 131.2 (*p*-CMe, C₆H₄Me-4), 137.1 $(CSO_2, C_6H_4Cl-4), 142.8 (p-CCl, C_6H_4Cl-4), 143.3$ (CS=N, C_6H_4 Me-4). Found, %: C 43.92; H 4.92; S 12.05; Sn 22.75. C₁₉H₂₆ClNO₂S₂Sn. Calculated, %: C 43.99; H 5.02; S 12.35; Sn 22.91.

4-Methylphenyl 3-(trimethylstannyl)propyl sulfone (I). To a solution of 5.84 g of sulfide II in 100 ml of MeOH we added at 0°C dropwise with stirring 3.62 g of 31% H_2O_2 . The reaction mixture was stirred for 3 h at 0°C and then for 6 h at room temperature, after which it was evaporated in a

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vacuum at room temperature. The residue was treated with 80 ml of warm hexane and filtered. The filtrate was reduced to 20 ml and cooled to -10° C to isolate 5.07 g (79%) of sulfone I as colorless crystals, mp 53–54°C (from hexane). IR spectrum, v, cm⁻¹: 1270, 1080 [SO₂ (as)], 1140 [SO₂ (s)], 765 (SnMe), 520 (SnC). ¹H NMR spectrum (CDCl₃), δ , ppm (*J*, Hz): 0.083 s (9H, Me₃Sn), d [Me₃Sn, *J*(H–¹¹⁷Sn) 51.2, *J*(H–¹¹⁹Sn) 53.5], 0.81 m (2H, SnCH₂), 1.92 m (2H, CH₂CH₂CH₂), 2.50 s (3H, C₆H₄CH₃-4), 3.10 m (2H, CH₂SO₂), 7.38–7.85 (AA'BB' system; 4H, C₆H· ₄CH₃-4). Found, %: C 43.58; H 6.28; S 8.77; Sn 32.20. C₁₃H₂₂O₂SSn. Calculated, %: C 43.24; H 6.14; S 8.88; Sn 32.38.

Lithium salt IX. A 0.6 N solution of BuLi in hexane (5.32 ml) was added with a syringe to a solution of 1.05 g of sulfone I in 15 ml of DMSO. A redbrown precipitate of lithium salt IX formed. The reaction mixture was shaken for 10 min and used in the reaction with methyl iodide.

Reaction of lithium salt IX with methyl iodide. Methyl iodide, 0.45 g, was added in a vacuum to the suspension of lithium salt IX, obtained as described above. The reaction mixture quickly decolorized. A 10% solution of NH_4Cl (20 ml) was then added, the organic layer was separated, the aqueous layer was extracted with ether $(3 \times 50 \text{ ml})$, the combined organic extracts were washed with water to neutral, dried with Na_2SO_4 , the solvent was removed, and the oily residue (1.00 g) was filtered on a glass filter and distilled in a vacuum to obtain 0.80 g (74%) of a mixture of isomeric sulfides **Xa** and **Xb** as a viscous liquid, bp 188–189°C (1.5 mm). IR spectrum, v, cm⁻¹: 1290, 1080 [SO₂ (as)], 1135 [SO₂ (s)], 760 (SnMe), 525 (SnC). Me₃SnCH₂CH₂CH(CH₃)SO₂C₆H₄CH₃-4 (Xa). ¹H NMR spectrum ($\tilde{C}DCl_3$), δ , ppm: 0.053 s (9H, CH₃Sn), 0.53–1.00 m (2H, SnCH₂), 1.27 d (3H, CHC H_3), 1.56 m and 2.17 m (2H, SnC H_2CH_2CH), 2.45 s (3H, C₆H₄CH₂), 2.90 m (1H, CHSO₂), 7.40-7.85 m (4H, $C_6H_4CH_3^3$). Me₃SnCH₂CH₂CH₂SO₂C₆H₄. $(CH_2CH_3)-4$ (Xb). ¹H NMR spectrum (CDCl₂), δ , ppm (J, Hz): 0.046 s (3H, CH₃Sn), 0.80 m (2H, SnCH₂), 1.27 t (3H, C₆H₄CH₂CH₃), 1.89 m (2H,

CH₂CH₂CH₂), 2.75 q (2H, C₆H₄CH₂CH₃, J 7.5), 3.07 m (2H, CH₂SO₂), 7.40–7.85 m (C₆H₄C₂H₅).

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