A Chiral Stannate as an "Ansa" π-Arene Ligand in Rhodium Chemistry

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General Experimental

All manipulations were performed under an inert gas atmosphere of dried argon (desiccant P₄O₁₀, Granusic[®], J.T. Baker) in standard (Schlenk) glassware or by working in a glove box. All reaction flasks were heated prior to use using three evacuation-refill cycles. Solvents and solutions were transferred by canula-septa techniques. Solvents were dried according to standard methods and saturated with argon and stored over potassium mirror. $MeSi{SiMe_2NH[(R)-CHMePh]}_3^{[1]}$ was prepared analogous to its (S)-enantiomer. $[RhCl(C_2H_4)_2]_2^{[2]}$ and $[RhCl(COE)_2]_2^{[3]}$ were synthesized according to literature procedures. Rhodium precursors were dried over MgSO₄ as a solution in dichloromethane. The solutions were reduced in vacuo and the complexes were precipitated by adding hexane. [RhCl(PⁱPr₃)₂]₂^[4] was prepared in situ according to Werner et al.. All other reagents were commercially available and used as received. ¹H, ⁷Li, ¹³C, ²⁹Si, ³¹P and ¹¹⁹Sn-NMR spectra were recorded on a Bruker DRX 200, Avance II 400 or Avance III 600. NMR spectra are quoted in ppm relative to tetramethylsilane (¹H and ¹³C); ⁷Li, ²⁹Si, ³¹P and ¹¹⁹Sn-NMR data are listed in ppm relative to an external standard (⁷Li: LiCl_{ag}, ²⁹Si: Si(CH₃)₄, ³¹P: 85% H₃PO₄ and ¹¹⁹Sn: Sn(CH₃)₄). ¹H- and ¹³C-NMR-spectra were referenced internally using the residual protonated solvent peak (^{1}H) or the carbon resonance (^{13}C) . Infrared spectra were recorded on a Varian 3100 FT-IR spectrometer. Elemental analyses were carried out in the microanalytical laboratory of the chemistry department of Heidelberg.

MeSi{SiMe₂N[(*R*)-CHMePh]]₃SnLi (1). MeSi{SiMe₂NH[(*R*)-CHMePh]]₃ (2.000 g, 3.46 mmol) was dissolved in Et₂O and cooled to -78 °C. Then a 2.5 M solution of n-BuLi (4.6 ml, 11.5 mmol) in hexane was added. Afterwards it was warmed to room temperature and stirred for 2 h. Again the temperature was decreased to -78 °C and the reaction solution was transferred to solid SnCl₂ (695 mg, 3.72 mmol). The orange suspension was allowed to warm to room temperature within 18 h. The solvent was removed in vacuo and the product was extracted with hexane. After concentrating the solution the product was isolated by

crystallization at -30 °C. 2 was washed with hexane and dried in vacuo to yield a light yellow powder (1.140 g, 47 %). (Found: C, 53.42; H, 7.16; N, 5.85. Calc. for C₃₁H₄₈LiN₃Si₄Sn: C, 53.14; H, 6.90; N, 6.00%); v_{max}(KBr)/cm⁻¹ 3025w, 2958w, 2892w, 1491w, 1450w, 1400w, 1368w, 1245m, 1200w, 1120m, 1031w, 944m, 838s, 778s and 760s; ¹H-NMR (400 MHz, C_6D_6): $\delta = 7.72$ (d, ${}^{3}J_{HH} = 7.7$ Hz, 2H, Ph), 7.31 (t, ${}^{3}J_{HH} = 7.7$ Hz, 2H, Ph), 7.2 (d, ${}^{3}J_{HH} =$ 7.2 Hz, 2H, Ph), 7.12 (m, 1H, Ph), 7.08 – 6.95 (m, 5H, Ph), 6.94 – 6.86 (m, 1H, Ph), 6.62 (d, ${}^{3}J_{HH} = 7.2$ Hz, 2H, Ph), 5.04 (q, ${}^{3}J_{HH} = 6.6$ Hz, 1H, CHMePh), 4.48 (q, ${}^{3}J_{HH} = 6.8$ Hz, 1H, CHMePh), 4.33 (q, ${}^{3}J_{HH} = 6.8$ Hz, 1H, CHMePh), 1.90 (d, ${}^{3}J_{HH} = 6.8$ Hz, 9H, CH(CH₃)Ph), 1.45 (d, ${}^{3}J_{HH} = 6.8$ Hz, 9H, CH(CH₃)Ph), 1.29 (d, ${}^{3}J_{HH} = 6.8$ Hz, 9H, CH(CH₃)Ph), 0.62 (s, 6H, Si(CH₃)₂), 0.48 (s, 3H, Si(CH₃)₂), 0.40 (s, 3H, Si(CH₃)₂), 0.29 (s, 3H, Si(CH₃)₂), 0.08 (s, 3H, SiCH₃), -0.54 (s, 3H, Si(CH₃)₂); ⁷Li-NMR (155.4 MHz, C₆D₆): $\delta = 0.23$ (s); ¹³C-NMR $(100.6 \text{ MHz}, C_6D_6)$: $\delta = 150.3 \text{ (s, 1-Ph)}, 149.6 \text{ (s, 1-Ph)}, 149.5 \text{ (s, 1-Ph)}, 131.1 \text{ (s, 3,5-Ph)},$ 129.4 (s, 3,5-Ph), 128.6 (s, 2,6-Ph), ca. 127.9 (s, 3,5-Ph), 127.6 (s, 4-Ph), 126.6 (s, 4-Ph), 126.4 (s, 2,6-Ph), 126.4 (s, 2,6-Ph), 126.0 (s, 4-Ph), 58.9 (s, CHMePh), 56.5 (s, 2x CHMePh), 27.0 (s, CH(CH₃)Ph), 24.4 (s, CH(CH₃)Ph), 23.9 (s, CH(CH₃)Ph), 5.3 (s, Si(CH₃)₂), 5.0 (s, $Si(CH_3)_2$, 4.6 (s, $Si(CH_3)_2$), 3.9 (s, $Si(CH_3)_2$), 3.5 (s, $Si(CH_3)_2$), 3.4 (s, $Si(CH_3)_2$), -15.2 (s, SiCH₃); ²⁹Si-NMR (79.4 MHz, C₆D₆): $\delta = -5.6$ (s, SiMe₂), -5.9 (s, SiMe₂), -8.0 (s, SiMe₂), -97.8 (s, SiMe); ¹¹⁹Sn-NMR (149.1 MHz, C₆D₆): δ = 11.9 (s).

[MeSi{SiMe₂N[(*R*)-CHMePh]}₂Sn{SiMe₂N[(*R*)-CHMe-η⁶-Ph]}Rh(COE)] (2a). At -78 °C precooled toluene was added to a mixture of MeSi{SiMe₂N[(*R*)-CHMePh]}₃SnLi (1) (458 mg, 0.656 mmol) and [RhCl(COE)₂]₂ (235 mg, 0.328 mmol). The reaction mixture was stirred for 10 min before it was allowed to warm to room temperature. Insolubilities were removed by centrifugation. The solvent was distilled of in vacuo to yield a brown powder (543 mg, 92 %). (Found: C, 51.43; H, 6.86; N, 4.52. Calc. for C₃₉H₆₂N₃RhSi₄Sn: C, 51.65; H, 6.89; N, 4.63%); v_{max}(KBr)/cm⁻¹ 3061w, 3017w, 2925w, 2893m, 2802w, 1599w, 1486w, 1446m, 1415w, 1241m, 1196m, 1129m, 1070m, 1018m, 966s, 936m, 828s, 798s, 763s and 704m; ¹H-NMR (400 MHz, C₆D₆): δ = 7.76 (d, ³J_{HH} = 8.0 Hz, 2H, 2,6-*H*_{Ph}), 7.69 (d, ³J_{HH} = 8.0 Hz, 2H, 2,6-*H*_{Ph}), 7.31 (t, ³J_{HH} = 7.7 Hz, 2H, 3,5-*H*_{Ph}),), 7.30 (t, ³J_{HH} = 7.7 Hz, 2H, 3,5-*H*_{Ph}),), 7.20 (m, 2H, 4-*H*_{Ph}), 6.14 (d, ³J_{HH} = 6.3 Hz, 1H, 2,6-*H*_{η6-Ph}), 5.86 (d, ³J_{HH} = 6.3 Hz, 1H, 2,6-*H*_{η6-Ph}), 5.38 (t, ³J_{HH} = 6.2 Hz, 1H, 3,5-*H*_{η6-Ph}), 5.27 (d, ³J_{HH} = 6.1 Hz, 1H, 3,5-*H*_{η6-Ph}), 5.02 (q, ³J_{HH} = 7.1 Hz, 1H, CHMePh), 5.01 (q, ³J_{HH} = 7.1 Hz, 1H, CHMePh), 4.62 (t, ³J_{HH} = 6.2 Hz, 1H, 4-*H*_{η6-Ph}), 3.88 (q, ³J_{HH} = 6.2 Hz, 1H, CHMe-η⁶-Ph), 2.85 – 2.70 (m, 1H, CH_{COE}), 2.60 – 2.45 (m, 1H, CH_{COE}), 1.86 (d, ³J_{HH} = 6.9 Hz, 3H, CH(CH₃)Ph), 1.82 – 1.75 (m, 1H, CH_{2 COE}), 1.67 (d, ³J_{HH} = 6.9 Hz, 3H, CH(CH₃)Ph), 1.65 – 1.60 (m, 1H, CH_{2 COE}), 1.42 (d, ³J_{HH} = 6.1 Hz, 3H, CH(CH₃)-η⁶-Ph), 1.20 – 1.05 (m, 4H, CH_{2 COE}), 1.02 – 0.80 (m, 6H, CH_{2 COE}), 0.70 (s, 3H, Si(CH₃)₂), 0.68 (s, 3H, Si(CH₃)₂), 0.64 (s, 3H, Si(CH₃)₂), 0.57 (s, 3H, Si(CH₃)₂), 0.41 (s, 3H, Si(CH₃)₂), 0.39 (s, 3H, Si(CH₃)₂), 0.23 (s, 3H, SiCH₃); ¹³C-NMR (100.6 MHz, C₆D₆); δ = 151.0 (s, 1-C_{Ph}), 149.7 (s, 1-C_{Ph}), 128.1 (s, 3,5-C_{Ph}), 127.8 (s, 3,5-C_{Ph}), 127.8 (s, 2,6-C_{Ph}), 127.4 (s, 2,6-C_{Ph}), 126.1 (s, 4-C_{Ph}), 126.0 (s, 4-C_{Ph}), 124.5 (d, ¹J(¹⁰³Rh-¹³C) = 4.2 Hz, 1-C_{η6-Ph}), 94.7 (s, br, 3,5-C_{η6-Ph}), 102.2 (s, br, 3,5-C_{η6-Ph}), 95.3 (d, ¹J(¹⁰³Rh-¹³C) = 2.5 Hz, 2,6-C_{η6-Ph}), 94.7 (s, br, 4-C_{η6-Ph}), 92.5 (s, ¹J(¹⁰³Rh-¹³C) = 2.6 Hz, 2,6-C_{η6-Ph}), 57.7 (s, CHMePh), 57.5 (s, CHMePh), 54.4 (s, CHMe-η⁶-Ph), 34.6 (s, CH_{2 COE}), 32.3 (s, CH_{2 COE}), 27.5 (s, CH(CH₃)Ph), 27.1 (s, CH(CH₃)Ph), 26.6 (s, CH_{2 COE}), 26.4 (s, CH_{2 COE}), 25.8 (s, CH(CH₃)-η⁶-Ph), 25.6 (s, CH_{2 COE}), 7.1 (s, Si(CH₃)₂), 6.6 (s, Si(CH₃)₂), 6.2 (s, Si(CH₃)₂), 4.4 (s, Si(CH₃)₂), 4.2 (s, Si(CH₃)₂), 2.5 (s, SiMe₂), -4.4 (s, SiMe₂), -98.7 (s, SiMe₁); ¹¹⁹Sn-NMR (149.1 MHz, C₆D₆); δ = -34.3 (d, ¹J(¹¹⁹Sn-¹⁰³Rh) = 995 Hz).

 $[MeSi{SiMe_2N[(R)-CHMePh]}_2Sn{SiMe_2N[(R)-CHMe-\eta^6-Ph]}Rh(C_2H_4)]$ (2b). At -78 °C a solution of [RhCl(C₂H₄)₂]₂ (56 mg, 0.144 mmol) in toluene was added to a solution of MeSi{SiMe₂N[(R)-CHMePh]}₃SnLi (1) (200 mg, 0.285 mmol) in toluene. The reaction mixture was stirred for 30 min and then it was allowed to warm to room temperature. Insolubilities were separated by centrifugation. The centrifugate was concentrated in vacuo and storing at -30 °C yielded a brown powder (179 mg, 76 %). (Found: C, 47.82; H, 6.35; N, 4.92. Calc. for C₃₃H₅₂N₃RhSi₄Sn: C, 48.06; H, 6.36; N, 5.09%); v_{max}(KBr)/cm⁻¹ 3058w, 2957m, 2891m, 1599w, 1491m, 1448m, 1406w, 1365m, 1241s, 1195m, 1103s, 1066m, 944s, 833s, 780s, and 756s; ¹H-NMR (400 MHz, C_6D_6): $\delta = 7.71$ (d, ³J_{HH} = 7.6 Hz, 2H, 2,6-H_{Ph}), 7.67 (d, ${}^{3}J_{HH} = 7.6$ Hz, 2H, 2,6- H_{Ph}), 7.31 (t, ${}^{3}J_{HH} = 7.5$ Hz, 4H, 3,5- H_{Ph}),), 7.23 – 7.16 (m, 2H, 4- H_{Ph}), 6.23 (d, ${}^{3}J_{HH}$ = 6.1 Hz, 1H, 2,6- H_{n6-Ph}), 5.83 (d, ${}^{3}J_{HH}$ = 6.1 Hz, 1H, 2,6- H_{n6-Ph}), 5.37 (t, ${}^{3}J_{HH} = 6.0$ Hz, 1H, 3,5- $H_{\eta 6-Ph}$), 5.22 (d, ${}^{3}J_{HH} = 5.9$ Hz, 1H, 3,5- $H_{\eta 6-Ph}$), 5.00 (q, ${}^{3}J_{HH} =$ 6.7 Hz, 1H, CHMePh), 4.90 (q, ${}^{3}J_{HH} = 6.7$ Hz, 1H, CHMePh), 4.24 (t, ${}^{3}J_{HH} = 6.1$ Hz, 1H, $4-H_{\eta6-Ph}$), 4.94 (q, ³J_{HH} = 6.1 Hz, 1H, CHMePh), 2.33 – 2.16 (m, 4H, C₂H₄), 1.79 (d, ³J_{HH} = 6.8 Hz, 3H, CH(CH₃)Ph), 1.60 (d, ${}^{3}J_{HH} = 6.7$ Hz, 3H, CH(CH₃)Ph), 1.40 (d, ${}^{3}J_{HH} = 6.2$ Hz, 3H, CH(CH₃)-η⁶-Ph), 0.69 (s, 3H, Si(CH₃)₂), 0.67 (s, 3H, Si(CH₃)₂), 0.61 (s, 3H, Si(CH₃)₂), 0.59 (s, 3H, Si(CH₃)₂), 0.36 (s, 3H, Si(CH₃)₂), 0.32 (s, 3H, Si(CH₃)₂), 0.21 (s, 3H, SiCH₃); ¹³C-NMR (100.6 MHz, C₆D₆): δ = 150.8 (s, 1-C_{Ph}), 149.8 (s, 1-C_{Ph}), 128.0 (s, 3,5-C_{Ph}), 127.9 (s, 3,5-C_{Ph}), 127.8 (s, 2,6-C_{Ph}), 127.7 (s, 2,6-C_{Ph}), 127.4 (d, ${}^{1}J({}^{103}Rh-{}^{13}C) = 4.5$ Hz, 1-C_{η 6-Ph}), 126.2 (s, 4-C_{Ph}), 126.0 (s, 4-C_{Ph}), 100.4 (d, ${}^{1}J({}^{103}Rh-{}^{13}C) = 1.7$ Hz, 3,5-C_{η 6-Ph}), 98.8 (d, ${}^{1}J({}^{103}Rh-{}^{13}C) = 1.5$ Hz, 3,5-C_{η 6-Ph}), 96.4 (d, ${}^{1}J({}^{103}Rh-{}^{13}C) = 2.8$ Hz, 2,6-C_{η 6-Ph}), 94.6 (d, ${}^{1}J({}^{103}Rh-{}^{13}C) = 2.4$ Hz, 2,6-C_{η 6-Ph}), 94.5 (d, ${}^{1}J({}^{103}Rh-{}^{13}C) = 0.9$ Hz, 4-C_{η 6-Ph}), 58.3 (s, CHMePh), 57.6 (s, CHMePh), 53.6 (s, CHMe- η ⁶-Ph), 34.0 (d, ${}^{1}J({}^{103}Rh-{}^{13}C) = 14.2$ Hz, C₂H₄), 27.3 (s, CH(CH₃)Ph), 27.2 (s, CH(CH₃)Ph), 25.3 (s, CH(CH₃)- η ⁶-Ph), 7.2 (s, Si(CH₃)₂), 7.1 (s, Si(CH₃)₂), 6.2 (s, Si(CH₃)₂), 4.9 (s, Si(CH₃)₂), 3.6 (s, Si(CH₃)₂), 2.6 (s, Si(CH₃)₂), -15.2 (s, SiCH₃); 29 Si-NMR (79.4 MHz, C₆D₆): $\delta = -2.1$ (s, SiMe₂), -2.7 (s, SiMe₂), -3.2 (s, SiMe₂), -98.0 (s, SiMe); 119 Sn-NMR (149.1 MHz, C₆D₆): $\delta = -16.8$ (d, ${}^{1}J({}^{119}$ Sn- ${}^{103}Rh) = 944$ Hz).

 $[MeSi{SiMe_2N[(R)-CHMePh]}_2Sn{SiMe_2N[(R)-CHMe-\eta^6-Ph]}Rh(P^iPr_3)] (3). P^iPr_3$ (190 µl, 0.992 mmol) was added drop wise to a suspension of [RhCl(COE)₂]₂ (93 mg, 0.130 mmol) in pentane. After 10 min the solvent was distilled of in vacuo and the purple residue was washed with acetone. It was dried in vacuo before the solid was redissolved in toluene and added to a solution of $MeSi{SiMe_2N[(R)-CHMePh]}_3SnLi$ (1) (182 mg, 0.260 mmol) in toluene at -78 °C. After 15 min in the cold the reaction mixture was allowed to warm to room temperature. Insolubilities were removed by centrifugation and the brown solution was concentrated in vacuo. Storing at -30 °C yielded brown crystals (167 mg, 61 %), suitable for X-ray diffraction studies. (Found: C, 49.92; H, 7.30; N, 4.53. Calc. for C₄₀H₆₉N₃PRhSi₄Sn: C, 50.21; H, 7.27; N, 4.39 %); v_{max}(KBr)/cm⁻¹ 3055w, 2951w, 2927w, 2867w, 1491w, 1444m, 1411w, 1365w, 1041m, 827w, 748w and 700w; ¹H-NMR (400 MHz, C_6D_6): $\delta = 7.82$ (d, ${}^{3}J_{HH} = 7.8$ Hz, 2H, 2,6- H_{Ph}), 7.67 (d, ${}^{3}J_{HH} = 7.9$ Hz, 2H, 2,6- H_{Ph}), 7.38 (t, ${}^{3}J_{HH} = 7.8 \text{ Hz}, 2H, 3.5-H_{Ph}), 7.27 \text{ (t, }{}^{3}J_{HH} = 7.8 \text{ Hz}, 2H, 3.5-H_{Ph}), 7.16 \text{ (m, }{}^{3}J_{HH} = 7.3 \text{ Hz}, 2H, 3.5-H_{Ph})$ 4- H_{Ph}), 7.11 (m, ${}^{3}J_{HH} = 7.3$ Hz, 2H, 4- H_{Ph}), 6.43 (d, ${}^{3}J_{HH} = 6.1$ Hz, 1H, 2,6- H_{n6-Ph}), 5.67 (t, ${}^{3}J_{HH} = 6.0$ Hz, 1H, 3,5- H_{n6-Ph}), 5.40 (t, ${}^{3}J_{HH} = 6.1$ Hz, 1H, 3,5- H_{n6-Ph}), 5.35 (d, ${}^{3}J_{HH} = 5.9$ Hz, 1H, 2,6- H_{n6-Ph}), 5.17 (m, 1H, CHMePh), 4.75 (m, 1H, CHMePh), 4.70 (t, ${}^{3}J_{HH} = 5.9$ Hz, 1H, 4- H_{n6-Ph}), 3.89 (m, 1H, CHMe- η^{6} -Ph), 1.84 (d, ${}^{3}J_{HH}$ = 6.7 Hz, 3H, CH(CH₃)Ph), 1.81 (d, ${}^{3}J_{HH}$ = 7.3 Hz, 3H, CH(CH₃)Ph), 1.53 – 1.39 (m, 3H, P[CH(CH₃)₂]₃), 1.42 (d, ${}^{3}J_{HH} = 6.3$ Hz, 3H, $CH(CH_3)-\eta^6-Ph), 0.89 - 0.78 \text{ (m, 18H, P}[CH(CH_3)_2]_3), 0.84 \text{ (s, 3H, Si}(CH_3)_2), 0.75 \text{ (s, 3H, Si}(CH_3)_2), 0.75 \text{ (s, 3H, Si}(CH_3)_2), 0.75 \text{ (s, 2H, Si}(CH_3)_2), 0.7$ Si(CH₃)₂), 0.72 (s, 3H, Si(CH₃)₂), 0.64 (s, 3H, Si(CH₃)₂), 0.59 (s, 3H, Si(CH₃)₂), 0.25 (s, 3H, SiCH₃), 0.24 (s, 3H, Si(CH₃)₂); ¹³C-NMR (100.6 MHz, C₆D₆): δ = 151.3 (s, 1-C_{Ph}), 149.1 (s, 1-C_{Ph}), 128.2 (s, 2,6-C_{Ph}), 128.1 (s, 2,6-C_{Ph}), 127.7 (s, 3,5-C_{Ph}), 127.5 (s, 3,5-C_{Ph}), 126.0 (s, 4-C_{Ph}), 125.6 (s, 4-C_{Ph}), 120.5 (s, 1- C_{n6-Ph}), 97.3 (s, 3,5- C_{n6-Ph}), 95.5 (s, 2,6- C_{n6-Ph}), 94.5 (s, 2,6-C_{n6-Ph}), 93.7 (s, 3,5-C_{n6-Ph}), 88.8 (s, 4-C_{n6-Ph}), 59.5 (s, CHMePh), 57.3 (s, CHMePh), 53.6 (s, *C*HMe- η^6 -Ph), 29.7 (d, ¹J(³¹P-¹³C) = 20.2 Hz, P[*C*H(CH₃)₂]), 27.3 (s, CH(*C*H₃)Ph), 25.5 (s, CH(*C*H₃)- η^6 -Ph), 25.3 (s, CH(*C*H₃)Ph), 20.5 (P[CH(*C*H₃)₂]), 19.9 (P[CH(*C*H₃)₂]), 9.6 (s, Si(*C*H₃)₂), 7.9 (s, Si(*C*H₃)₂), 7.1 (s, Si(*C*H₃)₂), 6.5 (s, Si(*C*H₃)₂), 6.4 (s, Si(*C*H₃)₂), 1.7 (s, Si(*C*H₃)₂), -15.1 (s, Si*C*H₃); ²⁹Si-NMR (79.4 MHz, C₆D₆): δ = -2.8 (s, SiMe₂), -3.6 (s, SiMe₂), -5.6 (s, SiMe₂), -99.6 (s, SiMe); ³¹P-NMR (161.9 MHz, C₆D₆): δ = 68.0 (d, ¹J(¹⁰³Rh-³¹P) = 224 Hz, ²J(¹¹⁹Sn-³¹P) = 198 Hz, ²J(¹¹⁷Sn-³¹P) = 190 Hz, SnRh(\eta⁶-Ph)(*P*ⁱPr₃)); ¹¹⁹Sn-NMR (149.1 MHz, C₆D₆): δ = 30.0 (dd, ¹J(¹¹⁹Sn-¹⁰³Rh) = 1087 Hz, ²J(¹¹⁹Sn-³¹P) = 199 Hz).

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