**N,N-Diethyl-2-bromobenzylamine.**{superscript}N\textsuperscript{15}I\textsuperscript{I} 2-Bromobenzyl bromide (4.34 g, 17.4 mmol) and 6.1 mL of diethylamine (58.9 mmol, 3.40 equiv) were dissolved in 30 mL of toluene. The reaction was stirred at rt for 14 h (precipitate of Et\textsubscript{2}NH\textsubscript{2}Cl). The mixture was filtered and the filtrate was concentrated in vacuo and purified by a Kugelrohr distillation (95–110 °C, 0.05 mm Hg) to yield 3.81 g (15.7 mmol, 91%) of a pale yellow oil. \textsuperscript{1}H NMR (CDCl\textsubscript{3}, 300 MHz): δ 1.05 (t, J = 7.2 Hz, 6H), 2.57 (q, J = 7.2 Hz, 4H), 3.63 (s, 2H), 7.04–7.11 (m, 1H), 7.28 (td, J = 7.36, 1.29 Hz, 1H), 7.51 (dd, J = 7.91, 1.29 Hz, 1H), 7.53–7.58 (m, 1H). \textsuperscript{13}C\textsuperscript{(1)}H NMR (CDCl\textsubscript{3}, 75.4 MHz): δ 11.92 (CH\textsubscript{3}), 47.15 (CH\textsubscript{2}), 57.12 (CH\textsubscript{2}), 124.08 (C), 127.10 (CH), 127.89 (CH), 130.48 (CH), 132.41 (CH), 139.56 (C). MS (EI): M\textsuperscript{+} = 243.0454 (calc. for C\textsubscript{11}H\textsubscript{16}BrN = 243.0477).

**N,N-Diethyl-2-trimethylstannylbenzylamine.** N,N-Diethyl-2-bromobenzylamine (1.42 g, 5.88 mmol) was dissolved in 20 mL of THF. The solution was cooled to −78 °C and 2.87 M n-BuLi in hexanes (2.10 mL, 6.03 mmol) was slowly added. After 2 min Me\textsubscript{3}SnBr (1.45 g, 5.98 mmol) in 5 mL of THF was added. The solution was taken up in 1:1 ether/hexanes (40 mL), washed with water (2 x 200 mL) and brine (100 mL), dried (MgSO\textsubscript{4}), filtered, and the solvent evaporated. Purification by Kugelrohr distillation (120–130 °C, 0.02 mm Hg) gave 1.60 g (4.91 mmol, 84%) of a clear colorless liquid. Density = 1.21 g/mL. \textsuperscript{1}H NMR (CDCl\textsubscript{3}, 300 MHz): δ 0.24 (s, \textsuperscript{2}J\textsubscript{H-H\textsubscript{119Sn}} = 54.2 Hz, 9H), 0.94 (t, J = 7.17 Hz, 6H), 2.52 (q, J = 7.17 Hz, 4H), 3.57 (s, 2H), 7.18–7.30 (m, 3H), 7.41–7.63 (m, 1H). \textsuperscript{13}C\textsuperscript{(1)}H NMR (CDCl\textsubscript{3}, 75.4 MHz): δ −7.62 (CH\textsubscript{3}), 9.33 (CH\textsubscript{3}), 44.20 (CH\textsubscript{2}), 60.34 (CH\textsubscript{2}), 126.39 (CH), 128.19 (CH), 128.56 (CH), 136.52 (CH), 142.52 (C), 145.94 (C). MS (EI): M\textsuperscript{+} = 312.0767 (calc. for C\textsubscript{14}H\textsubscript{25}SnN = 312.0773).

**2-Iodobenzamide.**{superscript}[\textsuperscript{15}N] A solution of 99% isotopically-enriched \textsuperscript{15}NH\textsubscript{4}Cl (0.486 g, 8.91 mmol) in 5 mL of water was covered with benzene (5 mL) and cooled to 0 °C. A solution of NaOH (0.750 g, 18.75 mmol, 2.11 equiv) in 5 mL of water was added directly to the aqueous layer via a Pasteur pipette inserted through the benzene layer. Immediately, a solution of 2-iodobenzyl chloride (2.3734 g, 8.91 mmol, 1.00 equiv) in 40 mL of benzene was added to the top layer. The biphasic solution was stirred vigorously at rt overnight while a white solid precipitated out of solution. The solution was then cooled to 0 °C. The precipitate was filtered, washed with cold water (100 mL) and cold benzene (50 mL), and allowed to air dry overnight to yield 1.82 g (7.34 mmol, 82%) of a white crystalline solid; m.p. 182–183 °C. \textsuperscript{1}H NMR (CDCl\textsubscript{3}, 300 MHz): δ 5.80 (d, \textsuperscript{1}J\textsubscript{H-15N} = 88.6 Hz, 1H), 5.81 (d, \textsuperscript{1}J\textsubscript{H-15N} = 89.7 Hz, 1H), 7.13 (ddd, J = 8.09, 7.35, 1.84 Hz, 1H), 7.40 (td, J = 7.72, 1.10 Hz, 1H), 7.49 (dd, J = 7.72, 1.84 Hz, 1H), 7.90 (dd, J = 8.09, 1.10 Hz, 1H). \textsuperscript{13}C\textsuperscript{(1)}H NMR (CDCl\textsubscript{3}, 75.4 MHz): δ 92.02 (C), 128.27 (CH; d, \textsuperscript{3}J\textsubscript{13C-15N} = 16.7 Hz), 131.46 (CH), 140.18 (CH), 141.11 (C; d, \textsuperscript{3}J\textsubscript{13C-15N} = 8.0 Hz), 170.85 (C; d, \textsuperscript{3}J\textsubscript{13C-15N} = 16.0 Hz). IR (KBr): 3353, 3175, 1641 cm\textsuperscript{-1}. MS (EI): M\textsuperscript{+} = 247.9461 (calc. for C\textsubscript{7}H\textsubscript{4}Cl\textsuperscript{15}NO = 247.9466).

**N,N-Diethyl-2-iodobenzamide.**{superscript}[\textsuperscript{15}N] Powdered KOH (3.315 g, 59.1 mmol, 8.05 equiv) was added to 7 mL of DMSO in a 25 mL round-bottom flask and stirred in rt for 15 min. A solution of 2.4 mL of EtI (30 mmol, 4.09 equiv) and 1.82 g of 2-iodobenzamide-\textsuperscript{[\textsuperscript{15}N]} (7.34 mmol) in 4 mL of DMSO was
added to the KOH solution. This reaction is very exothermic! Before the addition, the flask was placed in a rt bath to absorb any excess heat. The reaction mixture was poured into 100 mL of water and extracted with CH₂Cl₂ (4 x 20 mL). The organic extract was washed with water (3 x 50 mL) and brine (50 mL), dried over MgSO₄, filtered, and concentrated in vacuo to yield 2.13 g (7.01 mmol, 96%) of a clear, colorless oil. The crude product was clean by ¹H NMR and used further without purification. ¹H NMR (CDCl₃, 300 MHz): δ 0.70 (td, J = 6.99 Hz, 3H, Hδ), 2.58 Hz, 1H), 1.30 (td, J = 6.98 Hz, 3J_H-15N = 2.57 Hz, 3H), 3.14 (pentet, J = 6.99 Hz, 2H), 3.30 and 3.86 (ABX₃, J = 13.98, 6.99 Hz, 4H), 7.06 (td, J = 7.36, 1.48 Hz, 1H), 7.21 (dd, J = 7.73, 1.84 Hz, 1H), 7.38 (td, J = 7.35, 1.10 Hz, 1H), 7.82 (dd, J = 7.72, 1.10 Hz, 1H). ¹³C[¹H] NMR (CDCl₃, 75.4 MHz): δ 12.47 (CH₃), 13.90 (CH₃), 38.89 (CH₂; d, 3J_H-15N = 9.4 Hz), 42.75 (CH₂; d, 3J_H-15N = 10.2 Hz), 92.81 (C), 126.86 (CH), 128.22 (CH), 129.87 (CH), 139.15 (CH), 142.89 (C; d, 3J_H-15N = 7.3 Hz), 170.05 (C; d, 3J_H-15N = 16.0 Hz). IR (KBr): 3269, 1642 cm⁻¹. MS (EI): M⁺ = 304.0992 (calc. for C₁₁H₁₄NO = 304.0992).

N,N-Diethyl-2-iodobenzylamine-[¹⁵N]. N,N-Diethyl-2-iodobenzamide-[¹⁵N] (2.131 g, 7.01 mmol) was added to a dried and N₂-flushed 100 mL round-bottom flask equipped with a reflux condenser fitted with a septum. The flask was cooled to 0 °C and 1.0 M BH₃ in THF (15.0 mL, 15.0 mmol, 2.14 equiv) was added. The solution was heated to reflux for 5 h, cooled to 0 °C and 6M HCl (aq) (20.0 mL, 120 mmol, 17.1 equiv) was added dropwise over a period of 30 min. Caution: Addition of the 6M HCl (aq) too quickly will cause rapid H₂ gas evolution resulting in eruption of the solution and loss of material. The acidified solution was heated to reflux for 90 min (required to cleave the borane–amine complex), cooled to 0 °C and basified to pH 9 with NaOH pellets. The aqueous solution was mixed with 1:1 ether/hexanes (50 mL), the organic fraction was washed with water (2 x 200 mL) and brine (150 mL), dried over MgSO₄, filtered, and concentrated in vacuo to give a clear, colorless oil. Purification by Kugelrohr distillation (90–110 °C, 0.04 mm Hg) gave 1.85 g (6.39 mmol, 91%) of a clear, colorless oil.

¹H NMR (CDCl₃, 300 MHz): δ 1.04 (td, J = 6.99 Hz, 3J_H-15N = 2.58 Hz, 6H), 2.57 (q, J = 6.98 Hz, 4H), 3.56 (s, 2H), 6.92 (td, J = 7.72, 1.65 Hz, 1H), 7.30 (td, J = 7.36, 1.11 Hz, 1H), 7.51 (dd, J = 7.72, 1.47 Hz, 1H), 7.80 (dd, J = 7.90, 1.29 Hz, 1H). ¹³C[¹H] NMR (CDCl₃, 75.4 MHz): δ 11.96 (CH₃; d, 2J_H-15N = 1.91 Hz), 47.05 (CH₂; d, 3J_H-15N = 3.82 Hz), 62.25 (CH₂; d, 3J_H-15N = 5.08 Hz), 99.93 (C), 127.94 (CH), 128.27 (CH), 130.11 (CH, d, 3J_H-15N = 1.90 Hz), 139.13 (CH), 142.41 (C; d, 3J_H-15N = 3.18 Hz). MS (EI): M⁺ = 290.0305 (calc. for C₁₁H₁₄¹⁵N = 290.0300).

N,N-Diethyl-2-(trimethylstannyl)benzylamine-[¹⁵N]. Prepared from N,N-diethyl-2-iodobenzylamine-[¹⁵N] using the procedure for the unlabeled compound. The crude product was purified by Kugelrohr distillation (130–140 °C, 0.05 mm Hg) to yield 0.526 g (1.61 mmol, 45%) of a clear, colorless oil. Density = 1.21 g/mL. ¹H NMR (CDCl₃, 300 MHz): δ 0.24 (s, 3J_H-119Sn = 54.4 Hz, 9H), 0.94 (td, J = 7.35 Hz, 3J_H-15N = 2.21 Hz, 6H), 2.52 (q, J = 7.35 Hz, 4H), 3.57 (4J_H-119Sn = 5.0 Hz, 2H), 7.17–7.30 (m, 3H), 7.42–7.62 (m, 1H). ¹³C[¹H] NMR (CDCl₃, 75.4 MHz): δ -7.63 (CH₃), 9.34 (CH₂), 44.22 (CH₂; d, 3J_H-15N = 3.82 Hz), 60.35 (CH₂; d, 3J_H-15N = 4.45 Hz), 126.39 (CH), 128.18 (CH), 128.56 (CH), 136.52 (CH), 142.53 (C), 145.91 (C). ¹¹⁹Sn[¹H] NMR (THF, 134.3 MHz, -78 °C): δ -53.4 (d, 3J_H-119Sn-15N = 15.3 Hz). HRMS (ESI): M⁺ = 313.0777 (calc. for C₁₁H₁₄¹⁵N-Sn-CH₃ = 313.0743).

N-Isopropyl-N-methyl-2-bromobenzylamine. A solution of 2-bromobenzyl bromide (5.97 g, 23.9 mmol) and methylisopropylamine (5 mL, 48.0 mmol) in 30 mL of toluene was stirred at rt for 19 h. The reaction mixture was taken up in 1:1 ether/hexanes (30 mL), washed with water (150 mL) and brine (80 mL), dried over MgSO₄, filtered, and concentrated in vacuo. Kugelrohr distillation (70–80 °C, 0.03 mm Hg) gave 5.13 g (21.2 mmol, 89%) of a clear, colorless oil. ¹H NMR (CDCl₃, 300 MHz): δ 1.09 (d, J = 6.62 Hz, 6H), 2.19 (s, 3H), 2.94 (septet, J = 6.62 Hz, 1H), 3.60 (s, 2H), 7.04–7.12 (m, 1H), 7.24–7.31 (m, 1H), 7.47–7.54 (m, 2H). ¹³C[¹H] NMR (CDCl₃, 75.4 MHz): δ 17.99 (CH₃), 36.67
(CH₃), 53.71 (CH), 57.14 (CH₂), 124.38 (C), 127.13 (CH), 128.00 (CH), 130.59 (CH), 132.54 (CH), 139.30 (C). HRMS (EI): \( M^+ = 243.0444 \) (calc. for \( C_{14}H_{16}BrN = 243.0447 \)).

**N-Isopropyl-N-methyl-2-(trimethylstannyl)benzylamine.** \( N\)-Isopropyl-N-methyl-2-bromobenzylamine (0.751 g, 3.1 mmol) and 20 mL of THF was added to a dried and \( N_2 \)-flushed 50 mL round-bottom flask fitted with a septum. The flask was cooled to \(-78 \) °C and 2.87 M \( n \)-BuLi in hexanes (1.20 mL, 3.44 mmol) was added, the yellow solution was stirred at \(-78 \) °C for 10 min and 0.757 g of \( Me_3SnBr \) (3.11 mmol) in 7 mL of THF was added. Normal workup (1:1 ether/hexanes, water, brine) gave a yellow oil which was purified by a Kugelrohr distillation (140–150 °C, 0.03 mm Hg) to yield 0.765 g (2.35 mmol, 76%) of a clear, colorless oil. Density = 1.22 g/mL. \(^1\)H NMR (300 MHz, CDCl₃): \( \delta 0.24 \) (s, \( 2J^_{\text{HH}} = 54.1 \) Hz, 9H), 1.04 (d, \( J = 6.62 \) Hz, 6H), 1.88 (s, 3H), 2.98 (septet, \( J = 6.62 \) Hz, 1H), 3.57 (s, 2H), 7.15–7.34 (m, 3H), 7.41–7.63 (m, 1H). \(^{13}\)C\(^{\text{[H]}}\) NMR (75.4 MHz, CDCl₃): \( \delta -7.74 \) (CH₃), 17.13, 33.29 (CH₃), 53.51 (CH₂), 62.37 (CH₂), 126.45 (CH), 128.19 (CH), 128.79 (CH), 136.64 (CH), 142.07 (CH), 146.04 (CH). \(^{119}\)Sn\(^{\text{[H]}}\) NMR (THF, 134.3 MHz, \(-78 \) °C): \( \delta -54.0 \). MS (EI): \( M^+ -CH₃ = 312.0771 \) (calc. for \( C_{14}H_{23}NSn - CH₃ = 312.0777 \)).

**2-Bromoisoamylbenzene.** 1-Bromo-2-methyl-propane (6.33 g, 46.2 mmol) in 15 mL of THF was slowly added to Mg\(^\circ\) turnings (1.13 g, 46.5 mmol) in THF. The solution was refluxed until the Mg\(^\circ\) turnings disappeared. 2-Bromobenzyl bromide (8.63 g, 34.6 mmol) in 15 mL of THF was added over 10 min, and the solution refluxed for 6 h. The mixture was cooled to 0 °C and quenched slowly with 40 mL of 6M HCl. The solution was taken up in 1:1 ether/hexanes (60 mL), and the organic fraction washed with water and brine, and dried (MgSO₄). Solvent was evaporated and the residue purified by two Kugelrohr distillations (50–60 °C, 0.05 mm Hg) to yield 1.10 g (4.84 mmol, 14%) of a clear colorless oil. \(^1\)H NMR (300 MHz, CDCl₃): \( \delta 0.97 \) (d, \( J = 6.62 \) Hz, 6H), 1.44–1.54 (m, 2H), 1.65 (nonet, \( J = 6.62 \) Hz, 1H), 2.68–2.76 (m, 2H), 6.98–7.07 (m, 1H), 7.15–7.30 (m, 2H), 7.51 (d, \( J = 8.09 \) Hz, 1H). \(^{13}\)C\(^{\text{[H]}}\) NMR (75.4 MHz, CDCl₃): \( \delta -22.20 \) (CH₃), 28.03 (CH), 34.15 (CH₂), 39.21 (CH₂), 124.39 (C), 127.28 (CH), 127.34 (CH), 130.17 (CH), 132.74 (CH), 142.36 (C). HRMS (EI): \( M^+ = 228.0323 \) (calc. for \( C_{14}H_{23}Br = 228.0338 \)).

**2-(Trimethylstannyl)isoamylbenzene.** Prepared from 2-bromoisoamylbenzene using the procedure for \( N,N\)-diethyl-2-(trimethylstannyl)benzylamine. The crude product was purified by a Kugelrohr distillation (90–110 °C, 0.05 mm Hg) to yield 0.849 g (2.73 mmol, 70%) of a clear, colorless oil. \(^1\)H NMR (300 MHz, CDCl₃): \( \delta 0.31 \) (s, \( 2J^_{\text{HH}} = 54.1 \) Hz, 9H), 0.96 (d, \( J = 6.8 \) Hz, 6H), 1.43–1.54 (m, 2H), 1.66 (nonet, \( J = 6.8 \) Hz, 1H), 2.57–2.66 (m, 2H), 7.08–7.31 (m, 3H), 7.42 (dd, \( J = 7.3, 1.4 \) Hz; \( 2J^_{\text{HH}} = 57.0 \) Hz, 1H). \(^{13}\)C\(^{\text{[H]}}\) NMR (75.4 MHz, CDCl₃): \( \delta -8.15 \) (CH₃), 22.65 (CH₃), 28.43 (CH), 37.17 (CH₂), 41.84 (CH₂), 125.19 (CH), 128.07 (CH), 128.66 (CH), 136.12 (CH), 141.48 (C), 149.82 (C). \(^{119}\)Sn\(^{\text{[H]}}\) NMR (THF, 134.3 MHz, \(-78 \) °C): \( \delta -34.4 \). MS (EI): \( M^+ = 312.0923 \) (calc. for \( C_{14}H_{23}Sn = 312.0900 \)).

**3-Methoxybenzamide-[\(^{15}\)N].** 3-Methoxybenzonic acid (1.29 g, 8.5 mmol) was dissolved in \( CH_2Cl_2 \) (25 mL). A catalytic amount of dimethylformamide and thionyl chloride (0.674 mL, 9.35 mmol) were added, and the mixture refluxed overnight. The solvent was distilled and the residue taken up in benzene (15 mL). In a second flask, \(^{15}\)NH₄Cl (0.5 g, 9.18 mmol) was dissolved in 5 mL water and benzene (5.0 mL) was added. The flask was cooled to 0 °C, and 5.3 M NaOH (4.0 mL, 21.25 mmol) was carefully pipetted into the aqueous layer. The acid chloride solution was added and the reaction stirred overnight. The white solid was filtered, washed with water (70 mL) and benzene (15 mL) to give an 80% yield (1.03 g, 6.8 mmol). \(^1\)H NMR (CDCl₃, 300 MHz): \( \delta 7.43-7.30 \) (Ar-2, 4, & 6, m, 3H), \( \delta 7.07 \) (Ar-3, dt, \( J = 5.7 \) Hz (meta), \( J = 13.5 \) Hz (para), 1H), \( \delta 5.87 \) (NH₂, d, \( J = 120.5 \) Hz, 2H), \( \delta 3.87 \) (OMe, s). \(^{13}\)C NMR (CDCl₃, 90.56 MHz) \( \delta 55.9 \) (OMe), 113.0 (2-C), 118.7 (6-C) 119.6 (4-C), 130.0 (5-C), 135.2 (3-C, 5-C).
$^2J_{13C-15N} = 8.3$ Hz), 160.3 (1-C), 169.6 (C=O), $^1J_{13C-15N} = 15.5$ Hz). MS (EI): $M^+ = 152.0599$ (calc. for C$_8$H$_8$O$_2$N$_5$ = 152.0604).

$N,N$-Dimethyl-3-methoxybenzamide-[15N]. Powdered KOH (3 g, 53.4 mmol) was suspended in DMSO (10.5 mL) and 3-methoxybenzamide-[15N] (1.0 g, 6.7 mmol) and MeI (1.8 mL, 29.0 mmol) were added. The mixture was stirred at rt for 4 h. Water (100 mL) was added and the mixture extracted CH$_2$Cl$_2$ (4 x 15 mL). The organic fractions were washed with water (3 x 30 mL), brine (30 mL), dried (Na$_2$SO$_4$) and the solvent evaporated to yield (1.78 g, 6.21 mmol, 100%). $^1$H NMR (CDCl$_3$, 300 MHz): $\delta$ 7.33-7.28 (Ar-CH, m, 1H), d 6.98-6.91 (Ar-CH, m, 3H), d 3.87 (OMe, s), d 3.04 (CH$_3$, d, J = 76.5 Hz, 6H). $^1$C NMR (CDCl$_3$, 75.4 MHz) $\delta$ 35.0 (NMe), $^1J_{13C-15N} = 10.2$ Hz), 39.3 (NMe, $^1J_{13C-15N} = 10.8$ Hz), 55.1 (OMe), 112.2 (2-C), 115.2 (6-C) 118.9 (4-C), 129.2 (5-C), 137.5 (3-C, $^2J_{13C-15N} = 7.6$ Hz), 159.3 (1-C), 171.1 (C=O, $^1J_{13C-15N} = 15.9$ Hz). MS (EI): $M^+ = 180.0902$ (calc. for C$_{10}$H$_{12}$O$_2$N$_5^+$ = 180.0917).

$N,N$-Dimethyl-3-methoxybenzylamine-[15N]. To $N,N$-dimethyl-3-methoxybenzamide-[15N] (1.7 g, 9.5 mmol) at 0°C was added 1.0 M borane (15 mL, 15.0 mmol) in THF. The solution was refluxed for 4 h, quenched with 1 M HCl (3 mL), cooled to 0°C and 6 M HCl (10 mL) was added dropwise. The solution was again refluxed for 90 min to hydrolyze the borane-amine complex, cooled to 0°C, neutralized with NaOH pellets until the mixture was biphasic. Diethyl ether/hexanes (1:1, 25 mL) was added and the organic layer washed with 10% NaOH (8 x 35 mL), dried (Na$_2$SO$_4$), and the solvent evaporated (no heating). Kugelrohr distillation yielded (0.593 g, 3.56 mmol, 38.2%) of a colorless liquid. $^1$H NMR (CDCl$_3$, 300 MHz): $\delta$ 7.25-7.19 (Ar-CH, m, 1H), d 6.89-6.77 (Ar-CH, m, 3H), d 3.82 (OMe, s), d 3.42 (CH$_3$, s), d 2.24 (CH$_3$, s, 6H).

$N,N$-Dimethyl-3-methoxy-2-(trimethylstannyl)benzylamine-[15N]. To $N,N$-dimethyl-3-methoxybenzylamine-[15N] (0.55 g, 3.33 mmol) in THF at -78°C was added n-BuLi (2.6 mL, 6.7 mmol). The reaction mixture was stirred for 1.5 h, and HMPA (2.13 mL, 13.3) was added. The reaction was stirred for an additional 10 min, Me$_3$SnCl (1.0 g, 5.0 mmol) in THF was added by cannula. After 10 min the solution was warmed to rt, diluted with 50 mL Et$_2$O and washed with water (2 x 200 mL) and brine (150 mL), dried (Na$_2$SO$_4$), the solvent evaporated, and the product purified by Kugelrohr distillation (155°C, 0.3 mm) to yield 459 mg (1.26 mmol, 38.0%) of a clear colorless liquid. $^1$H NMR (CDCl$_3$, 300 MHz): $\delta$ 7.25-7.19 (Ar-CH, m, 1H), 6.89-6.77 (Ar-CH, m, 3H), 3.82 (OMe, s), 3.42 (CH$_3$, s), 2.24 (CH$_3$, s, 6H). $^1$C NMR (CDCl$_3$, 75.4 MHz) $\delta$ 5.6 (Me$_2$Sn), 27.1 (NMe, $^1J_{13C-15N} = 10.8$ Hz), 29.0 (NMe, $^1J_{13C-15N} = 8.9$ Hz), 55.9 (OMe), 65.5 (CH$_2$N, $^1J_{13C-15N} = 9.5$ Hz), 108.3 (6-C) 122.3 (4-C), 129.4 (5-C), 130.3 (2-C), 147.7 (3-C), 164.6 (1-C). MS (EI): M-Me$^+$ = 315.0535 (calc. for C$_{12}$H$_{20}$SnO$_2$N$_5$ = 315.0539).

(Chloromethyl)dimethyldisilyl(dimethylphenylsilyl)methane. Magnesium (610 mg, 25 mmol) was activated by vigorous stirring in ether (10 mL). (Chloromethyl)dimethylphenylsiline was added dropwise and the solution refluxed until Mg was no longer visible. In a second flask chloro(chloromethyl)dimethyldisilane (3.3 mL, 20.0 mmol) was dissolved in ether (10 mL). The solution was cooled to 0°C, and the Grignard solution was added slowly by cannula. The ether was evaporated under N$_2$ and THF (20 mL) was added and the solution stirred for 18 h at rt. The solvent was evaporated and hexanes (100 mL) and water (100 mL) were added and the mixture neutralized with 1 M HCl. The organic layer was washed with water (2 x 30 mL), dried (Na$_2$SO$_4$), and the solvent evaporated to give 4.15 g (81% yield) of a colorless liquid (contains trimethylphenylsiline). $^1$H NMR (200.132 MHz, CDCl$_3$) $\delta$ = 0.05 (s, $^2J_{Si-H} = 6.5$ Hz, 6H), 0.12 (s, 2H), 0.33 (s, $^2J_{Si-H} = 6.5$ Hz, 6H), 2.65 (s, 2H), 7.2-7.3 (m, 3H), 7.43-7.57 (m, 2H).
Dimethyl(N-pyrrolidinomethyl)silyl(dimethylphenylsilyl)methane (precursor for 16).
Prepared using the procedure for dimethyl(N-pyrrolidinomethyl)silyl(phenylthio)methane using (chloromethyl)dimethyl(dimethylphenylsilyle)methane (4.51 g, 16.0 mmol) and pyrrolidine (7 mL). The product was purified by column chromatography (0.5 L, 95:5 hexanes / ethyl acetate followed by 0.5 L 90:5:5 hexanes / EtOAc / triethylamine) yielding (3.56 g, 12.2 mmol, 61 %). 1H NMR (200.132 MHz, CDCl3) δ 0.02 (s, 6H), 0.05 (s, 2H), 0.31 (s, 2J_{Si-H} = 6.6 Hz, 6H), 1.73 (m, 4H), 1.93 (s, 2J_{Si-H} = 5.2 Hz, 2H), 2.41 (m, 4H), 7.2-7.3 (m, 3H), 7.45-7.57 (m, 2H). 13C{1H} NMR (90.556 MHz, CDCl3) δ = -0.3 (4C, SiMe2), 1.3 (1C, J_{C-Si} = 109 Hz, SiCH2Si), 23.8 (2C, β-pyrrolidine), 48.8 (1C, SiCH2N), 58.0 (2C, α-pyrrolidine), 128.6 (p), 127.6 (2C, m), 133.2 (2C, o), 141.4 (i). MS (EI): M+ = 291.1835 (calc. for C16H26NSi2 = 291.1838).

Chloro(dimethylphenylsilyl)(trimethylsilyl)methane. To a magnetically stirred solution of (chloromethyl)dimethylphenylsilane (3.60 mL, 20.0 mmol) in 20 mL of THF at -78 °C under N2 was added s-BuLi (1.41 M in pentane, 14.2 mL, 30.7 mmol) and TMEDA (3.17 mL, 20 mmol). The reaction was stirred at -50 °C for 30 min [S32] and Me3SiCl (3 mL, 23.6 mmol) was added. After 5 min the reaction was quenched with water, 100 mL of hexane were added, the hexane phase was extracted 3x with water and dried over Na2SO4. After solvent evaporation (30 min at 0.1 mm Hg) the product was purified by chromatography on silica gel ((chloromethyl)dimethylphenylsilane Rf = 0.37, product, Rf = 0.42) to give 1.89 g (37%) of silane. 1H NMR (300.133 MHz, CDCl3) δ = 0.02 (s, 2J_{Si-H} = 6.6 Hz, 9H), 0.45 (s, 2J_{Si-H} = 6.6 Hz, 6H), 2.66 (s, 2J_{Si-H} = 7.0 Hz, 2H), 7.26-7.38 (m, 3H), 7.56-7.59 (m, 2H). MS (EI): M+ = 258.0810 (calc. for C12H21^35ClSi2 = 258.0870).

(Dimethylphenylsilyl)(tri-n-butylstannyl)(trimethylsilyl)methane. (Precursor for 17) A solution of n-Bu3SnLi [S3] was prepared by addition of n-Bu3SnH (1.92 mL, 7.2 mmol) to a solution of LDA prepared from i-Pr2NH (1.27 mL, 9.0 mmol) and 2.31 M n-BuLi (3.16 mL, 7.3 mmol) in THF (10 mL) at 0 °C. Chloro(dimethylphenylsilyl)(trimethylsilyl)methane was added and the reaction stirred for 1 h at rt. Hexanes (100 mL) and water (100 mL) were added and the mixture neutralized with 1 M HCl. The organic layer was washed with water (2 x 30 mL), dried (Na2SO4), and the solvent evaporated. Kugelrohr distillation (100-120 °C, 0.1 mm) gave 2.62 g (71 % yield) of a colorless liquid. 1H NMR (300.133 MHz, CDCl3) δ = -0.27 (s, 2J_{Si-H} = 6.4 Hz, 2J_{Sn-H} = 74 Hz, 1H), 0.00 (s, 2J_{Si-H} = 6.3 Hz, 9H), 0.318 (s, 3H), 0.322 (s, 3H), 0.73-0.93 (m, 15H), 1.22-1.38 (m, 12H), 7.26-7.33 (m, 3H), 7.48-7.51 (m, 2H). 13C{1H} NMR (90.556 MHz, THF/ether 3:2) δ = -3.4 (1J_{C-H} = 105 Hz, 1J_{C-Si} = 58 Hz, 1J_{C-Sn} = 113 Hz, CH), 1.5 (1J_{C-Si} = 52 Hz, 1J_{C-Sn} = small, SiMe), 2.6 (1J_{C-Si} = 51 Hz, 1J_{C-Sn} = 17.8 Hz, SiMe), 3.6 (3C, 1J_{C-Si} = 51 Hz, 1J_{C-Sn} = 12.7 Hz, SiMe2), 12.6 (3C, 1J_{C-Sn} = 324 Hz, o-Bu), 13.7 (3C, δ-Bu), 28.1 (3C, 1J_{C-Sn} = 64 Hz, γ-Bu), 29.9 (3C, 1J_{C-Sn} = 18.5 Hz, β-Bu), 128.2 (2C, m), 129.1 (p), 133.9 (2C, o), 143.0 (1J_{C-Sn} = 12.7 Hz, i). MS (EI): M+ = 455.1638 (calc. for C20H39Si3Sn = 455.1611).

Dimethyl(methoxymethyl)silyl(phenylthio)methane. (Precursor for 18). Dichlorodimethylsilane (10 mL, 80 mmol) was dissolved in THF (25 mL), cooled to -78 °C, and 0.5 M phenylthiomethylthiium (20 mL, 10.0 mmol) [S41] was added. The reaction was stirred for 10 min and warmed to rt. The solvent and excess silane were evaporated under N2, the residue was taken up in THF (25 mL), the solution was cooled to -78 °C, and 1.24 M methoxymethylthiium (8.1 mL 10.0 mmol, prepared from methoxymethyltributylstannane [S3, S5] was added. Hexanes (100 mL) and water (100 mL) were added and the water layer was extracted with hexanes (3 x 50 mL). The organic layer was washed with water (2 x 30 mL), dried (Na2SO4), and the solvent evaporated. Kugelrohr distillation (50-80 °C, 0.5 mm) gave (1.17 g, 52 % yield) of a colorless liquid. 1H NMR (200.132 MHz, CDCl3) δ = 0.20 (s, 2J_{Si-H} = 6.7 Hz, 6H), 2.26 (s, 2J_{Si-H} = 6.5 Hz, 2H), 3.22 (s, 2J_{Si-H} = 7.0 Hz, 2H), 7.07-7.32 (m, 5H). 13C{1H} NMR (90.556 MHz, CDCl3) δ = -4.8 (2C, 1J_{C-Si} = 47 Hz, SiMe2), 15.4 (1J_{C-Sn} = 46 Hz, SCH2Si), 63.3 (OMe), 65.4
(¹J_{Si-C} = 61 Hz, SiCH₂O), 124.6 (p), 126.1 (2C, m), 128.6 (2C, o), 139.9 (i). MS (EI): M⁺ = 226.9857 (calc. for C₁₁H₁₉OSSi = 226.0848).

**General Procedure for Preparation of Arylithium Reagents from Aryltrimethylstannanes.** The aryltrimethylstannane (0.4 mmol) in a dried and N₂-flushed 10 mm NMR tube was cooled to -78 °C and 1 mL of dry Me₂O (or ether, when noted) was condensed in. (Me₂O was an ideal solvent for this crystallization method, since the lithium/tin exchange was relatively fast and the resulting aryllithium reagents were not soluble in pure Me₂O.) The NMR tube was briefly removed from the -78 °C bath, warmed slightly, and shaken to dissolve the aryllstaninate. The addition of 1 equiv of n-Bu₆Li or Et₆Li to the NMR tube gave a homogenous yellow solution. The tube was stored overnight at -78 °C while the aryllithium reagent crystallized out of solution. The yellow supernatant was removed by cannula transfer and the crystals were washed with ether (2 x 0.5 mL) at -78 °C. The crystals were dissolved in a combination of THF and ether, by both warming the solvent mixture to rt and vigorously shaking the NMR tube, to give a clear, colorless solution of aryllithium reagent with concentrations ranging from 0.08 to 0.18 M. The NMR tube was returned to the -78 °C bath, and Me₂O was added if needed. The sample was ready for NMR investigation.

**[Li⁶]**-EXSY Experiment of 1. To a N₂-flushed 10mm NMR tube was added N,N-dimethylbenzylamine (150 µL, 1.0 mmol) and 1.0 mL of ether. The flask was cooled to -78 °C and 1.11 M n-Bu₆Li (1.0 mL, 1.11 mmol) was added, and the tube warmed to rt for 2 d during which time 1 crystallized. The solvent was removed via cannula and the crystals washed with ether (2 x 2 mL) and dissolved in THF (1.5 mL) and ether (1.0 mL), to give a 0.4 M clear colorless solution. The Li⁶-EXSY spectrum was acquired at -128°C using the following parameters: a sweep width of 6.25 ppm (f1 and f2); 256 increments and 4 scans per increment; mixing time of 0.05 s. The spectrum is shown in Fig. S-1.

![Figure S-1. ⁶Li EXSY exchange spectrum of 1 at -128 °C.](image-url)
Variable-Temperature Experiment of 1-[\textsuperscript{6}Li]. THF (1.5 mL) and ether (0.5 mL) were added to a dried and N\textsubscript{2}-flushed 10 mm NMR tube. The NMR tube was cooled to -78 °C and a solution of 1-[\textsuperscript{6}Li] in THF (0.92 M, 0.54 mL, 0.50 mmol) was added. Addition of Me\textsubscript{2}O (1.0 mL) gave an initial aryllithium concentration of 0.14 M. The variable temperature experiment was monitored by \textsuperscript{6}Li and \textsuperscript{13}C NMR spectroscopy. Spectra are shown in Fig. 6. Simulations utilized the exchange matrix in Fig. 7,\textsuperscript{151} and the results of those simulations are reported in Fig. 8 and Table S-1.

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HMPA Titration of 1-[\textsuperscript{6}Li]. The sample from the VT study above was titrated with HMPA (0, 0.5, 1, 2, and 3 equiv) and monitored by \textsuperscript{6}Li and \textsuperscript{31}P NMR spectroscopy. The probe temperature ranged from -133 to -140 °C during the experiment. Spectra are shown in Fig. 13. In this particular case, precipitation became a problem beyond 3 equiv of HMPA. The sample was quenched with Me\textsubscript{2}S\textsubscript{2} (95 μL) to yield N,N-dimethyl-2-(methylthio)benzylamine (0.154 g, 84%) as a clear colorless liquid.

Dilution Experiment of 1-[\textsuperscript{6}Li]. A solution of 1-[\textsuperscript{6}Li] (0.31 g, 2.20 mmol) in ether (1.2 mL), and THF (1.8 mL) was added to a dried and N\textsubscript{2}-flushed 10 mm NMR tube to give a clear, pale yellow 0.73 M solution. The variable temperature experiment was monitored by \textsuperscript{13}C NMR spectroscopy at selected temperatures. After the experiment 1.0 mL of the solution was diluted with ether (1.2 mL) and THF (1.8 mL) in a second dried and N\textsubscript{2}-flushed 10 mm NMR tube to give a clear, pale yellow 0.18 M solution, and the NMR experiment was repeated. Sample spectra are shown in Fig. 10. Simulations of the loss of \textsuperscript{6}Li;\textsuperscript{13}C coupling in the carbanion were carried out using the exchange matrix for random exchange (Fig. 9)\textsuperscript{154} and the results are presented in Fig. 8 and 10, and Table S-2.

TMEDA Titration of 1-[\textsuperscript{6}Li]. A 0.88 M solution of 1-[\textsuperscript{6}Li] (0.6 mL, 0.53 mmol) in THF was added to THF (0.8 mL), ether (0.5 mL), and Me\textsubscript{2}O (1.0 mL) in a N\textsubscript{2}-flushed 10 mm NMR tube at -78 °C to make an aryllithium concentration of 0.18 M. \textsuperscript{6}Li and \textsuperscript{13}C NMR spectra were acquired at 0, 1, 2, 3, 4, and 5 eq. of TMEDA. Spectra are shown in Fig. S-2. The probe temperature ranged from -120 and -130 °C during the experiment. The sample was quenched with Me\textsubscript{2}S\textsubscript{2} (70 μL) and the product analyzed by GC: N,N-dimethyl-2-(methylthio)benzylamine (13.58 min, 0.474 mmol); N,N-dimethylbenzylamine (protonated Li reagent) (6.37 min, 0.077 mmol). Therefore, 0.474 meq (0.237 mmol dimer) were used as the mmol of aryllithium for the equilibrium calculations shown in the results section. The mmol for each dimer (A, B, C, A-TMEDA) and TMEDA, based on integrations of \textsuperscript{6}Li spectra, are shown in Table S-3 (volume of sample = 3.0 mL).
Table S-2. Data from the Simulation of the $^{13}$C NMR Variable Temperature Study of 1 at two Concentrations ($J_{C,Li} = 7.7$ Hz).

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Figure S-2. $^{13}$C$[^{1}H]$ NMR spectra of a TMEDA titration of 0.16 M 1-$[^{6}$Li] in 3:2:1 THF/Me$_2$O/ether at -135 $^\circ$C.
Table S-3. Complexation of 1 with TMEDA in 50:33:17 THF/Me₂O/ether at -135 °C

1 + TMEDA → 1·TMEDA  K_{eq} = [1·TMEDA]/([1][TMEDA])

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<th>[1·TMEDA]</th>
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Crystal Structure of 1·TMEDA. N,N-Dimethylbenzylamine (1.74 g, 12.9 mmol) was dissolved in ether (5 mL) in a 25 mL Schlenk flask. The flask was N₂ purged and 2.22 M n-BuLi (7.0 mL, 15.5 mmol) was added. Within 14 h crystals had precipitated. The crystals were washed with ether (2 x 5 mL), and redissolved in ether (10 mL) and TMEDA (3 - 4 mL). Within 1-2 d, a few large transparent crystals of 1·TMEDA were present on the bottom of the flask. The crystals were submitted in this form (covered with the solution) for X-ray analysis. The structure is shown in Fig. 12. The X-ray data have been reported previously.[158]

PMDTA Titration and Variable Temperature Experiment of 1-[²⁷Li]. A 0.91 M solution of 1-[²⁷Li] (0.55 mL, 0.5 mmol) in THF was added to THF (1.0 mL), ether (0.5 mL), and Me₂O (1.5 mL) in a N₂-flushed 10 mm NMR tube at -78 °C to give a 0.14 M ArLi solution. ²⁷Li and ¹³C NMR spectra were acquired at 0, 1, 2, 3, 4, and 5 eq. of PMDTA. A variable temperature study was carried out with 4 equiv of PMDTA present. Spectra are shown in Fig. S-3. The probe temperature ranged from -120 and -130 °C during the experiment. The sample was quenched with Me₂S₂ (70 μL, 1.5 equiv) and analyzed by GC: N,N-dimethyl 2-(methylthio)benzylamine (0.34 mmol, 70%), N,N-dimethylbenzylamine (0.065 mmol, 14%).

Variable Temperature Experiment of 1 / PhLi (1:1 mixture). To a N₂-flushed 10 mm NMR tube at -78 °C was added a THF solution of 1 (1.1 M, 0.59 mL, 0.64 mmol), an ether solution of PhLi (1.92 M, 0.33 mL, 0.64 mmol),[156] THF (0.6 mL), ether (0.3 mL), and Me₂O (2.2 mL) to make an aryllithium concentration of 0.14 M. ¹³C and ⁷Li NMR spectra were acquired at selected temperatures. Spectra are shown in Fig. 15 and Fig. S-4.

Simulation of the Mixed Dimer spectra. The exchange matrix of Fig. 16 was used to simulate the ⁶Li NMR spectra produced in the above experiment.[155] This matrix incorporates the approximations described in the text of the paper. The rate constants obtained and simulated spectra are shown in Fig. 15, the rate data in Fig. 17 and Table S-4.
Figure S-3. Left panel: $^6$Li NMR spectra of a PMDTA titration of 0.14 M 1 in 3:3:1 THF/Me$_2$O/ether at -120 to -132 °C. Right panel: variable temperate $^6$Li NMR spectra of 1 with 4 eq. of PMDTA. The signals of the two diastereomers of the 1-A dimer PMDTA complex, and a possible signal of PMDTA-monomer (1-M-PMDTA) are indicated on the figure.

HMPA titration of 1-[Li]/Ph$_4$Li (1:0.63 mixture). Phenyltrimethylstannane (0.117 g, 0.49 mmol) and 2.17 M n-Bu$^6$Li (0.24 mL, 0.50 mmol) were added to a N$_2$-flushed 10 mm NMR tube at -78 °C, and kept at -78 °C overnight. The n-BuMe$_3$Sn was removed in vacuo and the Ph$_4$Li dissolved in Me$_2$O (1.8 mL) followed by addition of 0.88 M 1-[Li] (0.6 mL, 0.52 mmol), 0.4 mL of THF, and 0.3 mL of Et$_2$O giving a total aryllithium concentration of 0.32 M. $^6$Li and $^{31}$P NMR spectra were acquired at 0, 0.2, 0.4, 0.6, 1.0, and 1.5 equiv. of HMPA. Selected spectra taken at -135 °C are shown in Fig. S-5. The sample was quenched with Me$_2$S$_2$ (120 μL) and analyzed by GC: $N,N$-dimethyl-2-(methylthio)benzylamine (0.51 mmol, 90%), thioanisole (0.32 mmol 65%).
Figure S-4. $^{13}$C{$^1$H} NMR spectra of: a) PhLi in THF/ether (80:20); b) 1:1 mixture of 2-lithio-$N,N$-dimethylbenzylamine (0.2 M) and PhLi (0.2 M) in Me$_2$O/THF/Et$_2$O (70:20:10); c) 2-lithio-$N,N$-dimethylbenzylamine (0.16 M) in THF/Me$_2$O/Et$_2$O (50:33:17).

Table S-4. Data Obtained from Simulation of the Mixed Dimer Exchange (1/PhLi/13).

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NMR study of o-Tolyllithium. THF (2.5 mL) was added to a dried and N$_2$-flushed 10 mm NMR tube. The tube was cooled to −78 °C and 0.92 M o-tolyllithium in hexanes (0.63 mL, 0.50 mmol) was added to give a clear colorless solution, with an aryllithium concentration of 0.16 M. The sample was stored overnight at −78 °C. $^{13}$C{$^1$H} NMR (CDCl$_3$, 90.0 MHz): Monomer: δ 121.1 (C$_1$, C$_4$), 123.4 (C$_5$), 142.3 (C$_6$), 148.0 (C$_7$), 195.8 (C$_8$, br); Dimer: δ 121.5 (C$_3$, C$_6$), 124.3 (C$_7$), 144.6 (C$_8$), 150.8 (C$_9$), 186.5 (C$_1$, br). See Fig. 1 for the $^6$Li NMR spectrum.
Figure S-5. $^6$Li NMR spectra of a 100:63 mixture of 1 and PhLi in Me$_2$O/THF/Et$_2$O (60/30/10) in presence of 0, 0.2, 0.4, 0.6 equiv of HMPA. A small amount of tetraalkylstannane was present ($h$ = HMPA). The mono-HMPA complexes of the mixed dimer 13 of are marked 13-$h_1$ and 13-$h_{1N}$ (HMPA complexed to the chelated lithium).

**TMEDA and PMDTA Titrations of 5-[$^6$Li].** A solution of 2-(trimethylstannyl)isoamylbenzene (0.155 g, 0.50 mmol) in THF (1.8 mL), and ether (1.2 mL) was added to a dried and N$_2$-flushed 10 mm NMR tube. The tube was cooled to -78 °C and 0.79 M n-Bu$^6$Li in hexanes (0.63 mL, 0.50 mmol) was added to give a yellow solution. The sample was stored overnight at -20 °C. The NMR tube was cooled to -78 °C and Me$_2$O (1.0 mL) was added to give an aryllithium concentration of 0.11 M.

The TMEDA titration experiment was monitored by $^6$Li NMR spectroscopy. The NMR spectra were acquired at 0, 0.5, 1, 2 and 4 equiv of TMEDA at -141 and -126 °C. Spectra are shown in Fig. S-6. After the experiment was completed, the sample was quenched with Me$_3$SiCl (100 µL, 0.788 mmol) to give a 92% yield of 1-methyl-4-(2'-trimethylsilylphenyl)butane by $^1$H NMR integration against pentachloroethane (30.0 µL, 0.249 mmol) as a standard.

On a similar 0.14 M sample of 5-[$^6$Li] a PMDTA titration experiment was monitored by $^6$Li and $^{13}$C NMR spectroscopy with spectra acquired at -125 °C with 0, 0.25, 0.5, 0.75 and 1 equiv of PMDTA. Spectra are shown in Fig. S-7 and Fig. S-8. After the experiment was completed, the sample was quenched with Me$_3$SiCl as above (93% yield).
Figure S-6. TMEDA titration of 0.11 M 6Li in 45:25:30 THF/Me₂O/Et₂O at -126 °C and -141 °C (t = TMEDA). In the right panel the TMEDA-complexed and THF-complexed aryllithium signals are decoalesced.

Figure S-7. 6Li NMR spectra of a PMDTA titration of 0.14 M 5 in 3:2 THF/Et₂O at -125 °C.

Figure S-8. 13C{¹H} NMR spectra of 0.14 M 5 and 5-PMDTA in 3:2 THF/Et₂O at -125 °C.
Variable-Temperature Experiment of 2-[\textsuperscript{6}Li,\textsuperscript{15}N]. Using the general procedure with ether as crystallizing solvent, a sample of 2-[\textsuperscript{6}Li,\textsuperscript{15}N] was prepared from \(N,N\)-diethyl-2-(trimethylstannyl)benzylamine-\([\textsuperscript{15}N]\) (0.3 g, 0.9 mmol and 1.54 M \(n\text{-Bu}\textsuperscript{6}Li\) in hexanes (0.62 mL, 0.95 mmol). The aryllithium crystals were dissolved in THF (1.8 mL), ether (1.2 mL), and \(Me_2O\) (1.0 mL) to give a clear, colorless 0.08 M solution. The variable temperature experiment was monitored by \(\text{^6Li}\) NMR. Spectra are shown in Fig. 4.

HMPA Titration of 2-[\textsuperscript{6}Li,\textsuperscript{15}N]. Using the general procedure with ether as crystallizing solvent, a sample of 2-[\textsuperscript{6}Li,\textsuperscript{15}N] was prepared from \(N,N\)-diethyl-2-(trimethylstannyl)benzylamine-\([\textsuperscript{15}N]\) (0.349 g, 1.07 mmol) and 2.05 M \(n\text{-Bu}\textsuperscript{6}Li\) in pentane (0.53 mL, 1.09 mmol). The aryllithium crystals were dissolved in THF (1.8 mL) and ether (1.2 mL) to give a clear, colorless 0.18 M solution. The HMPA titration experiment was monitored by \(\text{^6Li}\) and \(\text{^31P}\) NMR spectroscopy. The spectra were acquired at 0, 0.5, 1, 1.5, 2, 3 and 5 equiv of HMPA at -120 °C. Spectra are shown in Fig. S-9. After the experiment, the sample was quenched with \(Me_2SiCl\) (145 \(\mu\)L, 1.14 mmol) to give a 96% yield of \(N,N\)-diethyl-2-(trimethylsilyl)benzylamine by integration of the \(\text{^1H}\) NMR spectrum containing pentachloroethane (32.3 \(\mu\)L, 0.268 mmol) as a standard.

Figure S-9. HMPA titration of 0.08 M 2-[\textsuperscript{6}Li,\textsuperscript{15}N] in 3:2 THF/ether at -120°C (\(h = \text{HMPA}\)).

HMPA Titration of 2-[\textsuperscript{6}Li] in 3:2 \(Me_2\)THF/Ether. Using the general procedure with ether as crystallizing solvent, a sample of 2-[\textsuperscript{6}Li] was prepared from \(N,N\)-diethyl-2-(trimethylstannyl)benzylamine (0.241 g, 0.74 mmol) and 0.50 mL of 1.54 M \(n\text{-Bu}\textsuperscript{6}Li\) in pentane (0.77 mmol, 1.04 equiv). The aryllithium crystals were dissolved in 1.8 mL of \(Me_2\)THF and 1.2 mL of ether to give a clear, colorless 0.12 M solution. The HMPA titration experiment was monitored by \(\text{^6Li}\) and \(\text{^31P}\) NMR spectroscopy. The \(\text{^6Li}\) and \(\text{^31P}\) NMR spectra were acquired at 0, 0.5, 1, 2 and 3 equiv of HMPA (1 equiv = 64 \(\mu\)L) at -127 to -130 °C. Spectra are shown in Fig. S-10.
Figure S-10 HMPA titration of 0.12 M 2-[\(^6\)Li] in 3:2 Me\(_2\)THF/ether at -127\(^\circ\) C (h = HMPA).

**HMPA Titration of 3.** Using the general procedure with ether as crystallizing solvent, a sample of 3 was prepared from N-isopropyl-N-methyl-2-(trimethylstannyl)benzylamine (0.165 g, 0.506 mmol) and 2.13 M n-BuLi in pentane (0.24 mL, 0.511 mmol). The aryllithium crystals were dissolved in THF (1.8 mL) and ether (1.2 mL) to give a clear, colorless 0.08 M solution. The HMPA titration experiment (0, 0.5, 1, 2, 3, 4, 5 and 8 equiv., \(-120 ^\circ\) C) was monitored by \(^7\)Li and \(^31\)P NMR spectroscopy. A sample spectrum is shown in Fig. 14, the complete HMPA titration in Fig. S-11.

Figure S-11. HMPA titration of 0.08 M 3 in 3:2 THF/ether at -120 to -125 \(^\circ\) C (h = HMPA).

**HMPA Titration of 4-[\(^{15}\)N].** In a dried N\(_2\)-flushed 10 mm NMR tube Et\(^6\)Li (23 mg 0.66 mmol) was dissolved in ether (1.2 mL). The solution was cooled to -78 \(^\circ\) C, and
$N,N$-dimethyl-3-methoxy-2-(trimethylstannyl)benzylamine-$[^{15}N]$ (0.165 g, 0.506 mmol) was added. The solution was warmed until the $\text{Et}^6\text{Li}$ dissolved. The tube was cooled to -78 °C and THF (1.8 mL) and was added to give a clear, colorless 0.22 M solution. The HMPA titration experiment was monitored by $^6\text{Li}$, $^{15}\text{N}$, $^{13}\text{C}$ and $^{31}\text{P}$ NMR spectroscopy. The spectra were acquired at 0, 1, 2, 3, and 4 equiv of HMPA. The probe temperature was -127 °C during the experiment. Spectra are shown in Fig. 5.

**Variable Concentration Study of 4.** In a dried N$_2$-flushed 10 mm NMR tube $\text{Et}^6\text{Li}$ (72 mg, 2.06 mmol) was dissolved in ether (1.2 mL). The solution was cooled to -78 °C, and $N,N$-dimethyl-3-methoxy-2-(trimethylstannyl)benzylamine (542 μL, 2.05 mmol) was added. The solution was warmed until the $\text{Et}^6\text{Li}$ dissolved. The tube was cooled to -78 °C and THF (1.8 mL) was added to give a clear, colorless 0.70 M solution. The experiment was monitored by $^6\text{Li}$, and $^{13}\text{C}$ NMR spectroscopy with the probe temperature of -127 °C. A 1.0 mL portion was then transferred to a second dried N$_2$-flushed 10 mm NMR tube and diluted with ether (1.2 mL) and THF (1.8 mL) to give a clear, colorless 0.17 M solution. Spectra were acquired and the solution was diluted again in a similar fashion to give a clear, colorless 0.044 M solution. The spectra were acquired a final time on this sample. Relative concentrations of monomer and dimer were measured by line-shape simulation$^{15}$ of the $^6\text{Li}$ NMR spectra. The results of the experiment are shown in Fig. S-12.

![Figure S-12. Log [monomer] vs. log [dimer] plot for 4 in 3:2 THF/ether solvent at -127 °C.](image)

**HMPA titration of 16 : 17 (1:1 mixture).** A solution of 16 was prepared by adding 1.8 M $t$-BuLi (0.36 mL, 0.65 mmol) in pentane to a solution of (pyrrolidinomethyl)dimethylsilyl(dimethylphenylsilyl)methane (189 mg, 0.65 mmol) in ether (1.2 mL), and THF (1.8 mL) at -78 °C in a dried and N$_2$-flushed 10 mm NMR tube. The metalation was carried out at -19 °C for 16 h. In a second similar tube a solution of 17 was prepared by treating (dimethylphenylsilyl)(tri-n-butylstannyl)(trimethylsilylmethyl) methane (339 mg, 0.66 mmol) in ether (1.2 mL), and THF (1.8 mL) with 2.0 M $n$-BuLi (0.33 mL, 0.66 mmol) in pentane at -78 °C. In a third tube half of each of the above solutions was mixed yielding an organolithium concentration of 0.20 M. $^7\text{Li}$, $^6\text{Li}$ and $^{31}\text{P}$ NMR spectra were acquired at 0, 0.25, 0.50, 0.75, 1.0, and 1.25 eq. of HMPA. Spectra are shown in Fig. S-13. The probe temperature was -113 °C during the experiment. The data in Table S-5 were taken from the integrations of the $^7\text{Li}$ NMR spectra.
Figure S-13. HMPA titration of a solution of a mixture of 16 (0.1 M) and 17 (0.1 M) in 3:2 THF/ether at -113 °C.

Table S-5. Competitive titration of 16 and 17 with HMPA

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\(h = \text{HMPA} \quad K_{eq} = [16][17-\(h_1\)]/[16-\(h_1\)][17]

**HMPA titration of 15 : 18 (1:1 mixture).** A solution of 15 and 18 (each 0.09 M) was prepared by metalating a solution of trimethyilsilyl(phenylthio)methane (66 mg, 0.33 mmol) and dimethyl(methoxymethyl)silyl(phenylthio)methane (68 mg, 0.30 mmol) in ether (1.2 mL), and THF (1.8 mL) at -78 °C with 1.5 M \(t\)-BuLi (0.42 mL, 0.63 mmol) in pentane. The NMR tube was warmed to -18 °C overnight. \(^7\)Li and \(^{31}\)P NMR spectra were acquired at -121 °C with 0, 0.2, 0.4, 0.6, 0.8, and 1.0 eq. of HMPA. The data in Table S-6 were calculated using the ratio of \%18-\(h_1\) / \%15 in the \(^{31}\)P NMR spectra, a 1 : 1 ratio of chelated and non-chelated species, and the mmols of HMPA present.
Table S-6. Competitive titration of 18 and 15 with HMPA

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\(^a\) h = HMPA. \(^b\) K\(_{eq}\) = [18][15-\(h^1\)]/[18-\(h^1\)][15]

X-Ray Crystal Structure of 3-B-(THF)\(_2\)

Sample Preparation. N-Isopropyl-N-methyl-2-(trimethylstannyl)benzylamine (204.0 mg, 0.63 mmol) was dissolved in ether (1.5 mL) in a 10 mL Schlenk flask. The flask was N\(_2\) purged and 2.56 M n-BuLi (0.25 mL, 0.64 mmol) and THF (0.5 mL) was added. Within 24 h a few large transparent crystals of 3 were present on the bottom of the flask. The crystals were submitted in this form (covered with the solution) for X-ray analysis.

Data Collection. A yellow air-sensitive crystal with approximate dimensions 0.42 x 0.35 x 0.21 mm\(^3\) was selected under oil under ambient conditions and attached to the tip of a glass capillary. The crystal was mounted in a stream of cold nitrogen at 173(2) K and centered in the X-ray beam by using a video camera.

The crystal evaluation and data collection were performed on a Bruker CCD-1000 diffractometer with Mo K\(_\alpha\) (\(\lambda = 0.71073\) Å) radiation and the diffractometer to crystal distance of 4.9 cm.

The initial cell constants were obtained from three series of \(\omega\) scans at different starting angles. Each series consisted of 20 frames collected at intervals of 0.3\(^\circ\) in a 6\(^\circ\) range about \(\omega\) with the exposure time of 10 seconds per frame. A total of 38 reflections was obtained. The reflections were successfully indexed by an automated indexing routine built in the SMART program. The final cell constants were calculated from a set of 2691 strong reflections from the actual data collection.

The data were collected by using the hemisphere data collection routine. The reciprocal space was surveyed to the extent of a full sphere to a resolution of 0.80 Å. A total of 6316 data were harvested by collecting three sets of frames with 0.3\(^\circ\) scans in \(\omega\) with an exposure time 60 sec per frame. These highly redundant datasets were corrected for Lorentz and polarization effects. The absorption correction was based on fitting a function to the empirical transmission surface as sampled by multiple equivalent measurements. \(^{5-6}\)

Structure Solution and Refinement. The systematic absences in the diffraction data were consistent for the space groups P1 and P.\(^{5-7}\) The \(E\)-statistics strongly suggested the centrosymmetric space group \(P\) that yielded chemically reasonable and computationally stable results of refinement. A successful solution by the direct methods provided most non-hydrogen atoms from the \(E\)-map. The remaining non-hydrogen atoms were located in an alternating series of least-squares cycles and difference Fourier maps. All non-hydrogen atoms were refined with anisotropic displacement coefficients. All hydrogen atoms were included in the structure factor calculation at idealized positions.
and were allowed to ride on the neighboring atoms with relative isotropic displacement coefficients. The dinuclear complex occupies a crystallographic inversion center.

The final least-squares refinement of 166 parameters against 3067 data resulted in residuals $R$ (based on $F^2$ for $l=20$) and $wR$ (based on $F^2$ for all data) of 0.0524 and 0.1497, respectively. The final difference Fourier map was featureless.

The ORTEP diagrams are drawn with 30% probability ellipsoids. Views of the dimer unit and the unit cell are shown in Fig. S-14 and S-15.

Figure S-14. Two views of the single crystal X-ray structure of 3-B·(THF)$_2$ (ORTEP). The H-atoms are omitted for clarity.

Figure S-15. The content of the asymmetric unit.
Table S-7. Crystal Data and Structure Refinement for rei02.

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Empirical with SADABS

0.9867 and 0.9736

Full-matrix least-squares on F$^2$

3067 / 0 / 166

1.013

R1 = 0.0524, wR2 = 0.1497

R1 = 0.0742, wR2 = 0.1616

0.243 and -0.156 e.Å$^{-3}$
Table S-8. Atomic Coordinates \((x \times 10^4)\) and Equivalent Isotropic Displacement Parameters \((\text{Å}^2 \times 10^3)\) for rei02. \(U(\text{eq})\) is Defined as one Third of the Trace of the Orthogonalized \(U^{ij}\) Tensor.

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Symmetry transformations used to generate equivalent atoms: #1 -x,-y,-z
Table S-10. Anisotropic Displacement Parameters (Å² x 10³) for rei02. The Anisotropic Displacement Factor Exponent Takes the Form: 

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Symmetry transformations used to generate equivalent atoms: #1 -x, y, z
Table S-11. Hydrogen Coordinates (x 10^4) and Isotropic Displacement Parameters (Å² x 10^3)
for rei02.

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' Inquiries about the X-ray crystallographic studies should be directed to this author.

Supplemental Material References

S-7. All software and sources of the scattering factors are contained in the SHELXTL (version 5.1) program library (G. Sheldrick, Bruker Analytical X-Ray Systems, Madison, WI).