Electronic Supporting Information

Facile ring-opening of THF at lithium induced by a pendant Si-H bond and BPh₃

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General remarks
All manipulations were performed under argon atmosphere using standard Schlenk or glove box techniques. Prior to use, glasswares were dried overnight at 130 °C and solvents were dried, distilled and degassed using standard methods. (Me₃TA CD)H,S¹ Me₄TACD,S² [M{N(SiHMe₂)₂}],S³ and B(C₆F₅)₃S⁴ were synthesized following literature procedures. BPh₃ (95%) was purchased from abcr and purified by sublimation. NMR measurements were performed on a Bruker DRX 400 at ambient temperature unless otherwise mentioned. The chemical shifts (δ ppm) in the ¹H and ¹³C{¹H} NMR spectra were referenced to the residual proton signals of the deuterated solvents and reported relative to tetramethylsilane.S⁵ Abbreviations for NMR spectra: s (singlet), d (doublet), t (triplet), sep (septet), br (broad), IR spectra were measured on KBr pellets using an AVATAR 360 FT-IR spectrometer. Abbreviations for IR spectra: w (weak), m (medium), s (strong), br (broad). Elemental analyses were performed on an elementar vario EL machine. X-ray diffraction data were collected on a Bruker APEX II diffractometer. Single crystal diffraction data of 3 and 7 are reported in crystallographic information file (cif) accompanying this document.

Synthetic Procedures and spectroscopic data for 1-5 and 7

[{{Me₃TACD)H}Li{N(SiHMe₂)₂}}] (1)

A mixture of [Li{N(SiHMe₂)₂}] (0.100 g, 0.718 mmol) and LH (0.154 g, 0.718 mmol) in n-pentane (5 mL) was stirred for 0.5 h at room temperature. Removal of the volatiles under reduced pressure afforded analytically pure 1 (0.240 g, 0.679 mmol, 95%) as a colorless powder. ¹H NMR (400 MHz, benzene-d₆): δ 5.07 (sept, ¹J_{SiH} = 167 Hz, 2 H, SiHMe₂), 2.36-2.24 (m, 2 H, CH₂), 2.25 (s, 6 H, NMe), 2.19 (s, 3 H, NMe), 1.93-1.74 (m, 14 H, CH₂), 1.37
(br, m, 1 H, NH), 0.59 (d, $^3J_{HH} = 3.0$ Hz, 12 H, SiHMe$_2$). $^{13}$C{$_1^1$H} NMR (100 MHz, benzene-$d_6$): $\delta$ 56.0 (CH$_2$), 54.5 (CH$_2$), 53.4 (CH$_2$), 45.2 (CH$_2$), 44.4 (NMe), 44.4 (NMe), 7.3 (SiHMe$_2$). $^{29}$Si{$_1^1$H} NMR (80 MHz, benzene-$d_6$): $\delta$ –28.5. IR (KBr, cm$^{-1}$): 2039 (s, $\nu_{SiH}$).

Anal. Calcd. for C$_{15}$H$_{40}$N$_5$Si$_2$Li: C, 50.90; H, 11.40; N, 19.80. Found: C, 50.45; H, 11.15; N, 19.69.

Figure S1. $^1$H NMR spectrum of 1 in benzene-$d_6$.

Figure S2. $^{13}$C{$_1^1$H} NMR spectrum of 1 in benzene-$d_6$. 
Figure S3. $^{29}\text{Si}^{{\text{1}}\text{H}}$ NMR spectrum of $\mathbf{1}$ in benzene-$d_6$.

Figure S4. $^7\text{Li}^{{\text{1}}\text{H}}$ NMR spectrum of $\mathbf{1}$ in benzene-$d_6$.

Figure S5. Solid-state IR (KBr) spectrum of $\mathbf{1}$.
A mixture of $[\text{Na}\{(\text{N(SiHMe}_2)_2\})]$ (0.029 g, 0.187 mmol) and LH (0.040 g, 0.187 mmol) in $n$-pentane (5 mL) was stirred for 0.5 h at room temperature. Removal of the volatiles under reduced pressure afforded analytically pure 2 (0.065 g, 0.176 mmol, 94%) as a colorless powder. $^1$H NMR (400 MHz, benzene-$d_6$): $\delta$ 5.37 (sept, $^1J_{\text{SiH}} = 164$ Hz, 2 H, SiHMe$_2$), 2.17 (br, m, 2 H, CH$_2$), 2.10 (s, 6 H, NMe), 2.07 (s, 3 H, NMe), 1.87-1.59 (br, m, 14 H, CH$_2$), 0.69 (br, s, 1 H, NH), 0.61 (d, $^3J_{\text{HH}} = 2.8$ Hz, 12 H, SiHMe$_2$). $^{13}$C($^1$H) NMR (100 MHz, benzene-$d_6$): $\delta$ 55.4 (CH$_2$), 53.9 (CH$_2$), 53.2 (CH$_2$), 44.5 (CH$_2$), 44.1 (NMe), 44.0 (NMe), 7.4 (SiHMe$_2$). $^{29}$Si($^1$H) NMR (80 MHz, benzene-$d_6$): $\delta$ –31.9. IR (KBr, cm$^{-1}$): 1990 (s, $\nu_{\text{SiH}}$). Anal. Calcd. for C$_{15}$H$_{40}$N$_5$Si$_2$Na: C, 48.74; H, 10.91; N, 18.94. Found: C, 48.56; H, 10.43; N, 19.22.

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**Figure S6.** $^1$H NMR spectrum of 2 in benzene-$d_6$.

**Figure S7.** $^{13}$C($^1$H) NMR spectrum of 2 in benzene-$d_6$. 
Figure S8. $^{29}$Si{$^{1}$H} NMR spectrum of 2 in benzene-$d_{6}$.

Figure S9. Solid-state IR (KBr) spectrum of 2.

[(Me$_{3}$TACD)H]K{N(SiHMe$_{2}$)$_{2}$} (3)

A mixture of [K{(N(SiHMe$_{2}$)$_{2}$)}] (0.040 g, 0.233 mmol) and LH (0.050 g, 0.233 mmol) in n-pentane (5 mL) was stirred for 0.5 h at room temperature. Removal of the volatiles under reduced pressure afforded analytically pure 3 (0.085 g, 0.220 mmol, 94%) as a colorless powder. $^{1}$H NMR (400 MHz, benzene-$d_{6}$): $\delta$ 5.34 (sept, $^{1}$J$_{SiH}$ = 161 Hz, 2 H, SiHMe$_{2}$), 2.26-2.22 (m, 4 H, CH$_{2}$), 2.09 (s, 6 H, NMe), 2.05 (s, 3 H, NMe), 1.98-1.91 (m, 12 H, CH$_{2}$), 1.21 (m, 1 H, NH), 0.49 (d, $^{3}$J$_{HH}$ = 2.8 Hz, 12 H, SiHMe$_{2}$). $^{13}$C{$^{1}$H} NMR (100 MHz, benzene-$d_{6}$): $\delta$ 55.7 (CH$_{2}$), 54.6 (CH$_{2}$), 53.7 (CH$_{2}$), 45.1 (NMe), 44.5 (NMe), 44.1 (CH$_{2}$), 7.0 (SiHMe$_{2}$).

$^{29}$Si{$^{1}$H} NMR (80 MHz, benzene-$d_{6}$): $\delta$ -33.5. IR (KBr, cm$^{-1}$): 2032 (v$_{SiH}$), 1964 (v$_{SiH}$). Anal. Calcd. for C$_{15}$H$_{40}$N$_{5}$Si$_{2}$K: C, 46.70; H, 10.45; N, 18.15. Found: C, 46.36; H, 10.17; N, 18.19.
Figure S10. $^1$H NMR spectrum of 3 in benzene-$d_6$.

Figure S11. $^{13}$C{$^1$H} NMR spectrum of 3 in benzene-$d_6$.

Figure S12. $^{29}$Si{$^1$H} NMR spectrum of 3 in benzene-$d_6$. 
Figure S13. Solid-state IR (KBr) spectrum of 3.

A solution of 1 (0.100 g, 0.283 mmol) in 0.5 mL of benzene-$d_6$ was heated to 60 °C for 12 h. Progress of the reaction was monitored time to time by $^1$H NMR spectroscopy. After completion, all the volatiles were removed under reduced pressure to obtain a colorless solid. The solid was recrystallized from a concentrated n-pentane solution at −35 °C overnight and dried under vacuum to give analytically pure 4 (0.086 g, 0.244 mmol, 86%) as a white powder. $^1$H NMR (400 MHz, benzene-$d_6$): $\delta$ 5.46 (sept, $^1J_{\text{SiH}} = 169$ Hz, 1 H, SiHMe$_2$), 3.47 (m, 2 H, CH$_2$), 2.30-2.26 (br, m, 2 H, CH$_2$), 2.14-2.07 (br, m, 11 H, NMe and CH$_2$), 1.92-1.70 (br, m, 10 H, CH$_2$), 0.50 (s, 6 H, SiCH$_3$), 0.48 (d, $^3J_{\text{HH}} = 2.8$ Hz, 6 H, SiHMe$_2$). $^{13}$C{$^1$H} NMR (100 MHz, benzene-$d_6$): $\delta$ 61.5 (CH$_2$), 56.8 (CH$_2$), 53.7 (CH$_2$), 50.5 (CH$_2$), 44.9 (NMe), 42.2 (NMe), 6.6 (SiHMe$_2$), 5.5 (SiMe$_2$). $^{29}$Si{$^1$H} NMR (80 MHz, benzene-$d_6$): $\delta$ −16.4 (SiMe$_2$), −35.2 (SiHMe$_2$). IR (KBr, cm$^{-1}$): 2000 (s, $\nu_{\text{SiH}}$). Anal. Calcd. for C$_{15}$H$_{38}$N$_5$Si$_2$Li: C, 51.24; H, 10.89; N, 19.92. Found: C, 50.96; H, 10.77; N, 19.69.
Figure S14. $^1$H NMR spectrum of 4 in benzene-$d_6$.

Figure S15. $^{13}$C{$^1$H} NMR spectrum of 4 in benzene-$d_6$.

Figure S16. $^{29}$Si{$^1$H} NMR spectrum of 4 in benzene-$d_6$. 
A solution of 2 (0.150 g, 0.406 mmol) in 0.5 mL of benzene-$d_6$ was heated to 80 °C for 48 h. Progress of the reaction was monitored time to time by $^1$H NMR spectroscopy. After completion, all the volatiles were removed under reduced pressure to obtain a light brown colored solid. The solid was recrystallized from a concentrated $n$-pentane solution at −35 °C overnight and dried under vacuum to give analytically pure 5 (0.095 g, 0.258 mmol, 64%) as a white powder. $^1$H NMR (400 MHz, benzene-$d_6$): δ 5.76 (sept, $^1$J$_{SH}$ = 167 Hz, 1 H, SiHMe$_2$), 3.21-3.15 (m, 2 H, CH$_2$), 2.27-2.21 (m, 2 H, CH$_2$), 2.14-2.08 (m, 2 H, CH$_2$), 2.0 (br, m, 8 H,
NMe and CH₂), 1.84-1.76 (m, 4 H, CH₂), 1.67-1.61 (m, 2 H, CH₂), 1.54-1.48 (m, 5 H, NMe and CH₂), 0.61 (d, JHH = 2.8 Hz, 6 H, SiHMe₂), 0.54 (s, 6 H, SiMe₂). ¹³C{¹H} NMR (100 MHz, benzene-d₆): δ 57.5 (CH₂), 54.4 (CH₂), 53.7 (CH₂), 48.1 (CH₂), 43.5 (NMe), 42.8 (NMe), 7.0 (SiHMe₂), 5.2 (SiMe₂). ²⁹Si{¹H} NMR (80 MHz, benzene-d₆): δ −19.0 (SiMe₂), −37.6 (SiHMe₂). IR (KBr, cm⁻¹): 2000 (s, νSiH). Anal. Calcd. for C₁₅H₃₈N₅Si₂Na: C, 49.00; H, 10.42; N, 19.05. Found: C, 48.67; H, 10.08; N, 19.26.

Figure S19. ¹H NMR spectrum of 5 in benzene-d₆.

Figure S20. ¹³C{¹H} NMR spectrum of 5 in benzene-d₆.
[Li{(Me3TACD)SiMe2N(SiMe2O\textsubscript{n}Bu)BPh\textsubscript{3}}] (7)

Slow n-pentane diffusion into a solution of BPh\textsubscript{3} (0.071 g, 0.293 mmol) and 4 (0.103 g, 0.293 mmol) in 1 mL of THF at −35 °C over two days precipitated a white solid. The solid was isolated by decantation and dried under vacuum to obtain analytically pure 7 (0.172 g, 0.258 mmol, 88%) as a white powder. X-ray quality single crystals were also obtained in a similar fashion. \textsuperscript{1}H NMR (400 MHz, benzene-\textit{d}_6): δ 7.69-7.66 (m, 6 H, o-Ph), 7.33-7.29 (m, 3 H, p-Ph), 7.27-7.23 (m, 6 H, m-Ph), 3.91 (m, 2 H, OCH\textsubscript{2}CH\textsubscript{2}CH\textsubscript{2}CH\textsubscript{3}), 3.47 (m, 2 H, CH\textsubscript{2}), 2.28-2.25 (m, 2 H, CH\textsubscript{2}), 2.18 (br, s, 6 H, NMe), 2.13 (br, s, 3 H, NMe), 2.10-2.06 (m, 2 H, CH\textsubscript{2}), 1.94-1.55 (br, m, 12 H, CH\textsubscript{2} and OCH\textsubscript{2}CH\textsubscript{2}CH\textsubscript{2}CH\textsubscript{3}), 1.02 (t, J\textsubscript{HH} = 7.3 Hz, 3 H, OCH\textsubscript{2}CH\textsubscript{2}CH\textsubscript{2}CH\textsubscript{3}), 0.52 (s, 6 H, SiMe\textsubscript{2}), 0.41 (s, 6 H, SiMe\textsubscript{2}). \textsuperscript{13}C\textsuperscript{1}H NMR (100 MHz, benzene-\textit{d}_6): δ 139.3 (Ph), 131.7 (Ph), 128.1 (Ph), 68.2 (OCH\textsubscript{2}CH\textsubscript{2}CH\textsubscript{2}CH\textsubscript{3}), 61.4 (CH\textsubscript{2}), 56.6
(CH₂), 53.5 (CH₂), 50.4 (CH₂), 44.7 (NMe), 42.5 (NMe), 36.8 (OCH₂CH₂CH₂CH₃), 20.4 (OCH₂CH₂CH₂CH₃), 14.9 (OCH₂CH₂CH₂CH₃), 5.6 (SiMe₂), 4.0 (SiMe₂). ¹¹B NMR (128 MHz, THF-d₈): δ 67.9 (br, s). ²⁹Si{¹H} NMR (80 MHz, THF-d₈): δ −18.6 (SiMe₂), −22.1 (SiMe₂). Anal. Calcd. for C₃₇H₆₁N₅O₂Si₂BLi: C, 66.74; H, 9.23; N, 10.52. Found: C, 66.66; H, 9.22; N, 10.67.

Figure S23. ¹H NMR spectrum of 7 in benzene-d₆.

Figure S24. ¹³C{¹H} NMR spectrum of 7 in benzene-d₆.
Figure S25. $^{11}$B NMR spectrum of 7 in benzene-$d_6$.  

Figure S26. $^{29}$Si{$^1$H} NMR spectrum of 7 in benzene-$d_6$.  

Figure S27. Solid-state IR (KBr) spectrum of 7.
An NMR-scale reaction between BPh₃ and 4 in THF-d₈ showed immediate completion of ring-opening within <5 min to give Li[(Me₃TACD)SiMe₂NSiMe₂{O(CD₂)₃CD₂H}BPh₃] (7-d₈). NMR spectroscopic characterization indicates the presence of free BPh₃. ¹H NMR (400 MHz, THF-d₈): δ 7.55-7.52 (m, 6 H, o-Ph), 7.34-7.32 (m, 9 H, m-Ph and p-Ph), 3.51-3.44 (m, 2 H, CH₂), 2.79-2.72 (m, 2 H, CH₂), 2.62-2.46 (m, 8 H, CH₂), 2.40 (s, 3 H, NMe), 2.34 (s, 6 H, NMe), 2.40-2.34 (m, 2 H, CH₂), 2.12-2.06 (m, 2 H, CH₂), 0.91-0.84 (m, 1 H, OCD₂CD₂CD₂CD₂H), 0.04 (s, 6 H, SiMe₂), –0.18 (s, 6 H, SiMe₂). ¹³C {¹H} NMR (100 MHz, THF-d₈): δ 147.0 (ipso-Ph), 138.1 (Ph), 130.0 (Ph), 128.0 (Ph), 61.9 (CH₂), 57.3 (CH₂), 54.3 (CH₂), 50.9 (CH₂), 45.0 (NMe), 42.8 (NMe), 5.2 (SiMe₂), 3.5 (SiMe₂). ¹¹B NMR (128 MHz, THF-d₈): δ 48.3 (br, s). ²⁹Si {¹H} NMR (80 MHz, THF-d₈): δ –18.5 (SiMe₂), –22.1 (SiMe₂).

Figure S28. ¹H NMR spectrum of 7-d₈ in THF-d₈.

Figure S29. ¹³C {¹H} NMR spectrum of 7-d₈ in THF-d₈.

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Figure S30. $^1$B NMR spectrum of 7-$d_8$ in THF-$d_8$. The borate peaks (1.2 and $-2.4$ ppm) of low intensities could from impurities or intermediates in the exchange process.

Figure S31. $^{29}$Si{H} NMR spectrum of 7-$d_8$ in THF-$d_8$.

**Variable temperature NMR spectroscopic analysis of 7 and 7-$d_8$**

NMR spectroscopic characterization of 7 and 7-$d_8$ in benzene-$d_6$ and THF-$d_8$, respectively suggests that the BPh$_3$ exists as a 'free' borane in solution at room temperature, contrary to the solid-state structure. 7-$d_8$ is generated in situ by mixing 4 and BPh$_3$ in 1:1 ratio in THF-$d_8$. A variable temperature NMR spectroscopic analysis of 7 in toluene-$d_8$ and of 7-$d_8$ in THF-$d_8$ shows that the species undergoes temperature-dependent reversible association/dissociation between the zwitterionic 7 and a mixture of neutral 7' and BPh$_3$. 
Figure S32. Variable temperature $^{11}$B NMR spectra of 7 in toluene-$d_8$.

Figure S33. Variable temperature $^7$Li{1H} NMR spectra of 7 in toluene-$d_8$. 
Figure S34. Variable temperature $^1$H NMR spectra of 7 in toluene-$d_8$. 
Figure S35. Variable temperature $^{11}\text{B}$ NMR spectra of a 1:1 mixture of 4 and BPh$_3$ in THF-$d_8$.

Figure S36. Variable temperature $^7\text{Li}[^1\text{H}]$ NMR spectra of a 1:1 mixture of 4 and BPh$_3$ in THF-$d_8$.
**Figure S37.** Variable temperature $^1$H NMR spectra of a 1:1 mixture of 4 and BPh$_3$ in THF-$d_8$.

**Kinetic plots for the transformation of 1 to 4**

The kinetic measurement for the conversion of 1 to 4 (dehydrogenative Si–N bond formation) was conducted by monitoring the reaction with $^1$H NMR spectroscopy. A 0.6 mL of benzene-$d_6$ solution containing 1 (0.020 M) and 1,3,5-trimethoxybenzene (0.042 M) as an internal standard) was taken in a Teflon-sealed NMR tube. The NMR tube was placed in an oil bath preheated to 75 °C. Progress of the reaction was examined by removing the tube from the oil bath time to time and measuring the $^1$H NMR spectrum at room temperature. No reaction at room temperature ensured that there is no elapsed time during the spectral acquisition. The concentrations of 1 and 4 were determined by comparison of corresponding integrated resonances to the known concentration of 1,3,5-trimethoxybenzene. A first order rate constant ($k$) was obtained by a non-weighted linear least square fit of the data to the first order rate law, $\ln[1] = \ln[1]_0 + kt$. A value of $k = 5.9 \pm 0.1 \times 10^{-3}$ min$^{-1}$ was derived from the slope.
Figure S38. Plot of $[1]$ vs. time (min) at 75 °C in benzene-$d_6$ which shows an exponential decay.

Figure S39. Plot of ln$[1]$ vs. time (min) at 75 °C in benzene-$d_6$. 
4. X-ray crystallography

Single-crystal X-ray diffraction measurements of 3 and 7 were performed on a Bruker AXS diffractometer equipped with an Incoatec microsource and an APEX area detector using MoKα radiation (λ = 0.71073 Å), multilayer optics and ω-scans. Temperature control was achieved with an Oxford cryostream 700. The SMART program was used for data collection and unit cell determination; processing of the raw data frame was performed using SAINT+, multi scan absorption corrections were applied with SADABS. Both compounds are extremely sensitive towards air and moisture and decompose quickly. The structures were solved by direct methods (SIR-92). The molecule of 3 shows crystallographic inversion symmetry. The packing of 7 contains THF molecules within the crystal lattice. Within the molecule of 3, the CH2 carbon atoms of the Me3TACD ligands C1 – C8 are disordered. The disorder could be modeled with split positions. Within the molecule of 7, the CH2 carbon atom C14 and the CH3 carbon atom C15 of the n-butyl group are disordered. This disorder could also be modeled with split positions. The Refinements were performed against F2 with the program SHELXL-2013 using all reflections. Hydrogen atoms were included as riding on calculated positions with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(non-H), except for the atoms bound to silicon (H1 and H2 in 3) that were localized in difference Fourier maps and refined in their position with isotropic displacement parameters $U_{iso}(H) = 1.2U_{eq}$(Si). All non-hydrogen atoms were refined anisotropically, except for the atoms C1-C8 in 3 and O3 as well as C42-C47 (of the solvent molecule thf in 7) that were refined with split positions. Refinement results are given in Table S1. Graphical representations were performed with the program DIAMOND. CCDC-1544769 (3), -1544770 (7) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.
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