Lithium Diisopropylamide: Oligomer Structures at Low Ligand Concentrations.

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Supporting Information

I. $^6$Li NMR spectra of 0.1 M [$^6$Li,$^{15}$N$i$]-Pr$_2$NLi in 3:2 pentane:toluene at varying temperatures.

II. $^{15}$N NMR spectra of 0.1 M [$^6$Li,$^{15}$N$i$]-Pr$_2$NLi in 3:2 pentane:toluene at varying temperatures.

III. $^6$Li and $^{15}$N NMR spectra of 0.1 M [$^6$Li,$^{15}$N$i$]-Pr$_2$NLi in 3:2 pentane:toluene with 0.25 equiv of oxetane.

IV. $^6$Li,$^{15}$N-heteronuclear multiple quantum correlation (HMQC) spectrum of 0.1 M [$^6$Li,$^{15}$N$i$]-Pr$_2$NLi in 3:2 pentane:toluene with 0.25 equiv of oxetane.

V. $^6$Li and $^{15}$N NMR spectra of 0.1 M [$^6$Li,$^{15}$N$i$]-Pr$_2$NLi in 3:2 pentane:toluene with 0.5 equiv of oxetane.

VI. $^6$Li,$^{15}$N-heteronuclear multiple quantum correlation (HMQC) spectrum of 0.1 M [$^6$Li,$^{15}$N$i$]-Pr$_2$NLi in 3:2 pentane:toluene with 0.5 equiv of oxetane.

VII. $^6$Li and $^{15}$N NMR spectra of 0.1 M [$^6$Li,$^{15}$N$i$]-Pr$_2$NLi in 3:2 pentane:toluene with 0.5 equiv of oxetane: $^{15}$N decoupling.

VIII. $^6$Li and $^{15}$N NMR spectra of 0.1 M [$^6$Li,$^{15}$N$i$]-Pr$_2$NLi in 3:2 pentane:toluene with 10 equiv and 1.25 equiv of oxetane.

IX. $^6$Li spectra of 0.1 M [$^6$Li,$^{15}$N$i$]-Pr$_2$NLi in 3:2 pentane:toluene with varying amounts of oxetane.

X. $^6$Li and $^{15}$N NMR spectra of 0.1 M [$^6$Li,$^{15}$N$i$]-Pr$_2$NLi in 3:2 pentane:toluene with 0.25 equiv of THF.

XI. $^6$Li,$^{15}$N-heteronuclear multiple quantum correlation (HMQC) spectrum of 0.1 M [$^6$Li,$^{15}$N$i$]-Pr$_2$NLi in 3:2 pentane:toluene with 0.25 equiv of THF.

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XII. $^6$Li and $^{15}$N NMR spectra of 0.1 M $[^6\text{Li},^{15}\text{N}]i\text{-Pr}_2\text{NLi}$ in 3:2 pentane:toluene with 0.25 equiv of THF: $^{15}$N decoupling.

XIII. $^6$Li and $^{15}$N NMR spectra of 0.1 M $[^6\text{Li},^{15}\text{N}]i\text{-Pr}_2\text{NLi}$ in 3:2 pentane:toluene with 0.25 equiv of THF.

XIV. $^6$Li,$^{15}$N-heteronuclear multiple quantum correlation (HMQC) spectrum of 0.1 M $[^6\text{Li},^{15}\text{N}]i\text{-Pr}_2\text{NLi}$ in 3:2 pentane:toluene with 0.75 equiv of THF.

XV. $^6$Li NMR spectra of 0.1 M $[^6\text{Li},^{15}\text{N}]i\text{-Pr}_2\text{NLi}$ in 3:2 pentane:toluene with varying amounts of THF.

XVI. $^6$Li and $^{15}$N NMR spectra of 0.1 M $[^6\text{Li},^{15}\text{N}]i\text{-Pr}_2\text{NLi}$ in 3:2 pentane:toluene with 0.25 equiv of diethyl ether.

XVII. $^6$Li,$^{15}$N-heteronuclear multiple quantum correlation (HMQC) spectrum of 0.1 M $[^6\text{Li},^{15}\text{N}]i\text{-Pr}_2\text{NLi}$ in 3:2 pentane:toluene with 0.25 equiv of diethyl ether.

XVIII. $^6$Li NMR spectra of 0.1 M $[^6\text{Li},^{15}\text{N}]i\text{-Pr}_2\text{NLi}$ in 3:2 pentane:toluene with varying amounts of diethyl ether.

XIX. $^6$Li NMR spectra of 0.1 M $[^6\text{Li},^{15}\text{N}]i\text{-Pr}_2\text{NLi}$ in 3:2 pentane:toluene with varying amounts of diisopropylamine.

XX. $^6$Li NMR spectra of 0.1 M $[^6\text{Li}]i\text{-Pr}_2\text{NLi}$ in 3:2 pentane:toluene with varying amounts of diisopropylamine.
Figure 1. $^6$Li NMR spectra of 0.1 M $[^6\text{Li},^{15}\text{N}]_2\text{-i-Pr}_2\text{Li}$ in 3:2 pentane:toluene: (A) $^6\text{Li}[^{15}\text{N}]$ spectrum at -135 °C; (B) $^6\text{Li}$ spectrum at -135 °C; (C) $^6\text{Li}[^{15}\text{N}]$ spectrum at -127 °C; (D) $^6\text{Li}$ spectrum at -127 °C; (E) $^6\text{Li}[^{15}\text{N}]$ spectrum at -110 °C; (F) $^6\text{Li}$ spectrum at -110 °C; (G) $^6\text{Li}[^{15}\text{N}]$ spectrum at -100 °C; (H) $^6\text{Li}$ spectrum at -100 °C.
Figure II. $^{15}$N NMR spectra of 0.1 M $[^6]$Li,$^{15}$N$i$-Pr$_2$NLi in 3:2 pentane:toluene: 
(A)$^{15}$N[$^1$H] spectrum at -127 °C; (B) $^{15}$N[$^1$H] spectrum at 
-100 °C.
Figure III. $^6$Li and $^{15}$N NMR spectra of 0.1 M $[^6$Li,$^{15}$N]$i$-$Pr_2$NLi in 3:2 pentane:toluene with 0.25 equiv of oxetane at -135 °C: (A) $^{15}$N spectrum; (B) $^6$Li spectrum; (C) $^{15}$N[$^1$H, $^6$Li] spectrum.
Figure IV. $^6$Li and $^{15}$N NMR spectra of 0.1 M $[^6$Li,$^{15}$N]$^i$-Pr$_2$NLi in 3:2 pentane:toluene with 0.5 equiv of oxetane at -135 °C; (A) $^6$Li$^{[15]$N}$ spectrum; (B) $^6$Li spectrum; (C) $^{15}$N$^{[1$H}, $^6$Li$]$ spectrum; (D) $^{15}$N$^{[1$H}$ spectrum.
Figure V. $^6$Li,$^{15}$N-heteronuclear multiple quantum correlation (HMOC)
spectrum of 0.1 M $[^6$Li,$^{15}$N]$^i$-Pr$_2$NLi in 3:2 pentane:toluene with 0.5 equiv of
oxetane at -135 °C.
Figure VI. $^6\text{Li}^{15}\text{N}$-heteronuclear multiple quantum correlation (HMQC) spectrum of 0.1 M $[^6\text{Li},^{15}\text{N}]\text{LDA}$ in 3:2 pentane:toluene with 0.25 equiv of oxetane at -135 °C.
Figure VII. $^6$Li and $^{15}$N NMR spectra of 0.1 M $[^6$Li,$^{15}$N]$i$-Pr$_2$NLi in 3:2 pentane:toluene with 0.5 equiv of oxetane at -135 °C: (A) $^6$Li spectrum; (B) $^{15}$N($^1$H, $^6$Li) spectrum; (C) $^6$Li spectrum, $^{15}$N single frequency decoupling at 77.4 ppm; (D) $^6$Li spectrum, $^{15}$N single frequency decoupling at 75.7 ppm; (E) $^6$Li spectrum, $^{15}$N single frequency decoupling at 71.0 ppm (Axis is displayed in Hz.); (F) $^6$Li spectrum, $^{15}$N single frequency decoupling at 66.0 ppm.
Figure VIII. $^6$Li and $^{15}$N NMR spectra of 0.1 M $[^6$Li,$^{15}$N]$^i$-Pr$_2$NLi in 3:2 pentane:toluene: (A) $^6$Li spectrum with 10 equiv oxetane at -120 °C; (B) $^6$Li spectrum with 1.25 equiv oxetane at -135 °C; (C) $^{15}$N($^1$H) spectrum with 1.25 equiv oxetane at -135 °C.
Figure IX. $^{6}\text{Li}^{15}\text{N}]$ NMR spectra of 0.1 M $[^{6}\text{Li},^{15}\text{N}]$LDA in 3:2 pentane:toluene at -135 °C with: (A) no added ligand; (B) 0.25 equiv oxetane; (C) 0.5 equiv oxetane; (D) 0.75 equiv oxetane; (E) 1.0 equiv oxetane.
Figure X. $^6\text{Li}$ and $^{15}\text{N}$ NMR spectra of 0.1 M $[^6\text{Li},^{15}\text{N}]i$-Pr$_2$NLi in 3:2 pentane:toluene with 0.25 equiv of THF at -135 ºC: (A) $^6\text{Li}[^{15}\text{N}]$ spectrum; (B) $^6\text{Li}$ spectrum; (C) $^{15}\text{N}[^{1}\text{H},^6\text{Li}]$ spectrum; (D) $^{15}\text{N}[^{1}\text{H}]$ spectrum.
Figure XI. $^6\text{Li}[^{15}\text{N}]$-heteronuclear multiple quantum correlation (HMQC) spectrum of 0.1 M $[^6\text{Li},^{15}\text{N}]$-Pr$_2$NLi in 3:2 pentane:toluene with 0.25 equiv of THF at -135 °C.
Figure XII. $^6$Li and $^{15}$N NMR spectra of 0.1 M [$^6$Li,$^{15}$N]$i$-Pr$_2$NLi in 3:2 pentane:toluene with 0.25 equiv of THF at -135 °C: (A) $^6$Li spectrum; (B) $^{15}$N[$^1$H, $^6$Li] spectrum; (C) $^6$Li spectrum, $^{15}$N single frequency decoupling at 83.0 ppm; (D) $^6$Li spectrum, $^{15}$N single frequency decoupling at 81.3 ppm; (E) $^6$Li spectrum, $^{15}$N single frequency decoupling at 76.6 ppm; (F) $^6$Li spectrum, $^{15}$N single frequency decoupling at 74.5 ppm; (G) $^6$Li spectrum, $^{15}$N single frequency decoupling at 72.3 ppm; (H) $^6$Li spectrum, $^{15}$N single frequency decoupling at 70.0 ppm.
Figure XIII. $^6$Li and $^{15}$N NMR spectra of 0.1 M [$^6$Li,$^{15}$N]$^3$-Pr$_2$NLi in 3:2 pentane:toluene with 0.5 equiv of THF at -135 °C: (A) $^6$Li[$^{15}$N] spectrum; (B) $^6$Li spectrum; (C) $^{15}$N[1H, $^6$Li] spectrum; (D) $^{15}$N[1H] spectrum.
Figure XIV. $^6$Li,$^{15}$N-heteronuclear multiple quantum correlation (HMOC) spectrum of 0.1 M [$^6$Li,$^{15}$N]-Pr$_2$NLi in 3:2 pentane:toluene with 0.75 equiv of THF at -135 °C.
Figure XV. $^6\text{Li}$ and $^{15}\text{N}$ NMR spectra of 0.1 M $[^6\text{Li},^{15}\text{N}]i$-Pr$_2$NLi in 3:2 pentane:toluene at -135 °C with: (A) 0.25 equiv THF; (B) 0.5 equiv THF; (C) 0.75 equiv THF; (D) 1.0 equiv THF.
Figure XVI. $^6$Li and $^{15}$N NMR spectra of 0.1 M $[^6$Li,$^{15}$N]$i$-$\text{Pr}_2\text{NLi}$ in 3:2 pentane:toluene with 0.25 equiv of diethyl ether at -127 °C: (A) $^6$Li($^{15}$N) spectrum; (B) $^6$Li spectrum; (C) $^{15}$N($^1$H, $^6$Li) spectrum; (D) $^{15}$N($^1$H) spectrum.
Figure XVII. $^6\text{Li}^{15}\text{N}$-heteronuclear multiple quantum correlation (HMQC) spectrum of 0.1 M [$^6\text{Li}^{15}\text{N}$]-Pr$_2$NLi in 3:2 pentane/toluene with 0.25 equiv of diethyl ether at -127 °C.
Figure XVIII. ²⁶Li NMR spectra of 0.1 M [²⁶Li,¹⁵N]⁻⁻Pr₂NLi in 3:2 pentane:toluene at -127 °C with: (A) 0.1 equiv diethyl ether; (B) 0.25 equiv diethyl ether; (C) 0.5 equiv diethyl ether; (D) 0.75 equiv diethyl ether; (E) 1.0 equiv diethyl ether.
Figure XIX. $^{13}$C NMR spectra of 0.1 M $[6\text{Li,}^{15}\text{N}]\text{Pr}_2\text{NLi}$ in 3:2 pentane:toluene with 2 equiv of diethyl ether at: (A) -75 °C; (B) -90 °C; (C) -115 °C; (D) -127 °C.
Figure XX. $^6\text{Li}$ NMR spectra of 0.1 M $i$-$\text{Pr}_2\text{NLi}$ in 3:2 pentane:toluene: (A) $^6\text{Li}^{[15\text{N}]}$ spectrum of $[\text{Li}^{[15\text{N}]}\text{i-Pr}_2\text{NLi}$ with no added ligand; (B) $^6\text{Li}^{[15\text{N}]}$ spectrum of $[\text{Li}^{[15\text{N}]}\text{i-Pr}_2\text{NLi}$ with 0.5 equiv diisopropylamine; (C) $^6\text{Li}^{[15\text{N}]}$ spectrum of $[\text{Li}^{[15\text{N}]}\text{i-Pr}_2\text{NLi}$ with 1.0 equiv diisopropylamine; (D) $^6\text{Li}$ spectrum of $[\text{Li}^{[15\text{N}]}\text{i-Pr}_2\text{NLi}$ with 2.0 equiv diisopropylamine; (E) $^6\text{Li}$ spectrum of $[\text{Li}^{[15\text{N}]}\text{i-Pr}_2\text{NLi}$ with 10 equiv diisopropylamine; (F) $^6\text{Li}$ spectrum of $[\text{Li}\text{i-Pr}_2\text{NLi}$ with 20 equiv diisopropylamine; (G) $^6\text{Li}^{[15\text{N}]}$ spectrum of $[\text{Li}^{[15\text{N}]}\text{i-Pr}_2\text{NLi}$ with 1 equiv diisopropylamine and 1 equiv THF.