Figure 31. $^6$Li NMR spectra showing R*OLi (10b), 3:1 RLi/R*OLi (15) mixed tetramers. Spectra were recorded on mixtures of [$^6$Li]LiCpA and [$^6$Li,$^{15}$N]10b (prepared in situ from the alcohol and 1.3 equiv. of [$^6$Li]LiHMDS(2)) in diethyl ether at −95 °C. The total titer of 6 and 10b is 0.1 M. Spectra B and D were recorded with $^{15}$N broad-band decoupling.
Figure 32. $^6$Li NMR spectra showing 2:2 RLi/R*OLi (16), 1:3 RLi/R*OLi (21) mixed tetramers. Spectra were recorded on mixtures of $[^6$Li]$^1$CPA and $[^6$Li,$^{15}$N]$^{10}$b (prepared in situ from the alcohol and 1.3 equiv. of $[^6$Li]LiHMDS(4)) in diethyl ether at $-95^\circ$C. The total titer of 6 and 10b is 0.1 M. Spectra B and D were recorded with $^{15}$N broad-band decoupling.
Figure 33. $^{15}$N NMR spectra showing 3:1 RLi/R*OLi (15), 2:2 RLi/R*OLi (16) and 1:3 RLi/R*OLi (21) mixed tetramers. Spectra were recorded on mixtures of $[^{6}$Li]$\text{LiCPA}$ and $[^{6}$Li,$^{15}$N]$^{10}$b (prepared in situ from the alcohol and 1.3 equiv. of $[^{6}$Li]$\text{LiHMDS}$(*)) in diethyl ether at $-95$ °C. The total titer of 6 and 10b is 0.1 M. Spectra B, D, and F were recorded with $^{6}$Li broad-band decoupling.
Figure 34. Spectrum of 3:1 RLi/R*OLi showing mixed tetramers 15 and 16. Spectra were recorded on mixtures of [6Li,13C]LiCPA and [6Li]10b (prepared in situ from the alcohol and 1.3 equiv. of [6Li]LiHMDS(∗)) in diethyl ether at −95 °C. The total titer of 6 and 10b is 0.1 M. (A) 6Li,13C-HMQC of 3:1 [6Li,13C]6/[6Li]10b.
Figure 35. Spectrum of 2:2 RLi/R*OLi showing mixed tetramers 16 and 21. Spectra were recorded on mixtures of [6Li,13C]LiCPA and [6Li]10b (prepared in situ from the alcohol and 1.3 equiv. of [6Li]LiHMDS(†)) in diethyl ether at −95 °C. The total titer of 6 and 10b is 0.1 M. (A) J-resolved spectrum of 2:2 [6Li,13C]6/[6Li]10b.
Figure 36. Spectra of 3:1 RLi/R*OLi showing mixed tetramers 15, 16, and 21. Spectra were recorded on mixtures of [6Li]LiCPA and [6Li,15N]10b (prepared in situ from the alcohol and 1.3 equiv. of [6Li]LiHMDS(∗)) in diethyl ether at −95 °C. The total titer of 6 and 10b is 0.1 M. (A) 6Li,15N-HMQC of 1:1 [6Li,15N]6/[6Li]10b; (B) 6Li J-resolved spectrum of 1:1 [6Li,15N]6/[6Li]10b.
Figure 37. $^6$Li NMR spectra showing a 1:2 mix of 6 and $[^6$Li]$^\text{LiHMDS}^\text{*})$. (A) $[^6$Li]$[^{13}$C]$\text{LiCPA}$ and $[^6$Li]$^\text{LiHMDS}^\text{*})$ with $^{13}$C broad band decoupling; (B) $[^6$Li]$[^{13}$C]$\text{LiCPA}$ and $[^6$Li]$^\text{LiHMDS}^\text{*})$; (C) $[^6$Li]$\text{LiCPA}$ and $[^6$Li]$[^{15}$N]$\text{LiHMDS}^\text{*})$. All spectra were recorded in DMEA at $-100$ °C.
Figure 38. $^{13}$C NMR spectra showing a 1:1 mix of 6 and [6Li]LiHMDS(*)). (A) [6Li,$^{13}$C]LiCPA and [6Li]LiHMDS(*) with 6Li decoupling; (B) [6Li,$^{13}$C]LiCPA and [6Li]LiHMDS(*). All spectra were in DMEA at −100 °C.
Figure 39. $^{15}$N NMR spectra showing a 1:2 mix of 6 and $[^6\text{Li},^{15}\text{N}]\text{LiHMDS}^(*)$). (A) $[^6\text{Li}]\text{LiCPA}$ and $[^6\text{Li},^{15}\text{N}]\text{LiHMDS}^(*)$ with $^6\text{Li}$ decoupling (B) $[^6\text{Li}]\text{LiCPA}$ and $[^6\text{Li},^{15}\text{N}]\text{LiHMDS}^(*)$). All spectra were recorded in DMEA at $-100$ °C.
Figure 40. Spectra of a 1:2 mix of 6 and $[^{6}\text{Li}]\text{LiHMDS}^{(*)}$). Spectra were recorded on mixtures of $[^{6}\text{Li},^{13}\text{C}]\text{LiCPA}$ and $[^{6}\text{Li}]\text{LiHMDS}^{(*)}$ in DMEA at $-100 \degree \text{C}$. (A) $^{6}\text{Li}$-resolved spectrum of 1:2 $[^{6}\text{Li},^{13}\text{C}]\text{LiCPA}/[^{6}\text{Li}]\text{LiHMDS}^{(*)}$). (B) $^{6}\text{Li},^{13}\text{C}$-HMOC of 1:2 $[^{6}\text{Li},^{13}\text{C}]\text{LiCPA}/[^{6}\text{Li}]\text{LiHMDS}^{(*)}$).
Figure 41. Spectra of a 1:2 mix of 6 and [6Li,\(^{15}\text{N}\)]LiHMDS(*). Spectra were recorded on mixtures of [6Li]LiCPA and [6Li,\(^{15}\text{N}\)]LiHMDS(\(^*\)) in DMEA at -100 °C. (A) J-resolved spectrum of 1:2 [6Li]LiCPA/[6Li,\(^{15}\text{N}\)]LiHMDS(\(^*\)). (B) 6Li,\(^{15}\text{N}\)-HMQC of 1:2 [6Li]LiCPA/[6Li,\(^{15}\text{N}\)]LiHMDS(\(^*\)).