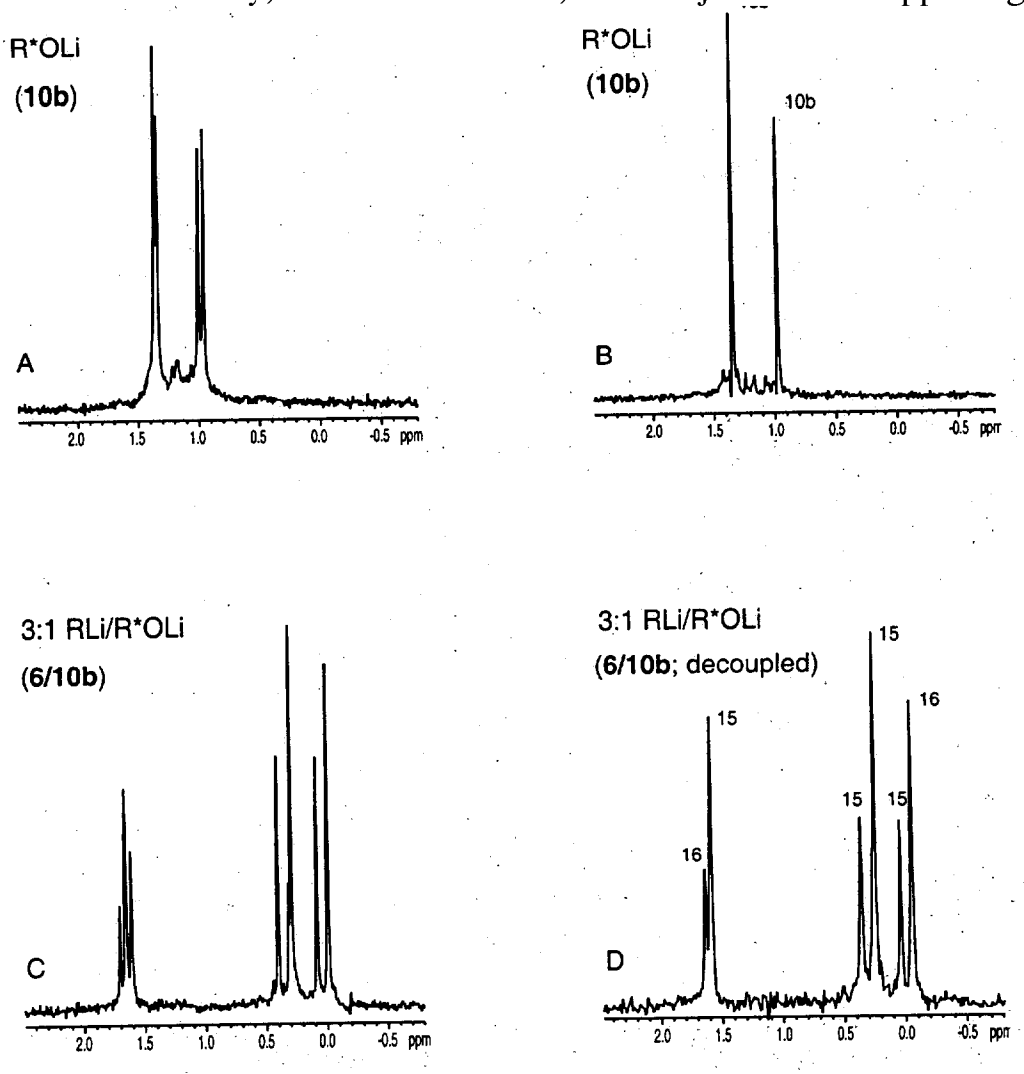

Figure 31. ^{6}Li NMR spectra showing R*OLi (**10b**), 3:1 RLi/R*OLi (**15**) mixed tetramers. Spectra were recorded on mixtures of [^{6}Li]LiCPA and [^{6}Li,^{15}N]**10b** (prepared in situ from the alcohol and 1.3 equiv. of [^{6}Li]LiHMDS(*)) in diethyl ether at –95 °C. The total titer of **6** and **10b** is 0.1 M. Spectra B and D were recorded with ^{15}N broad-band decoupling.

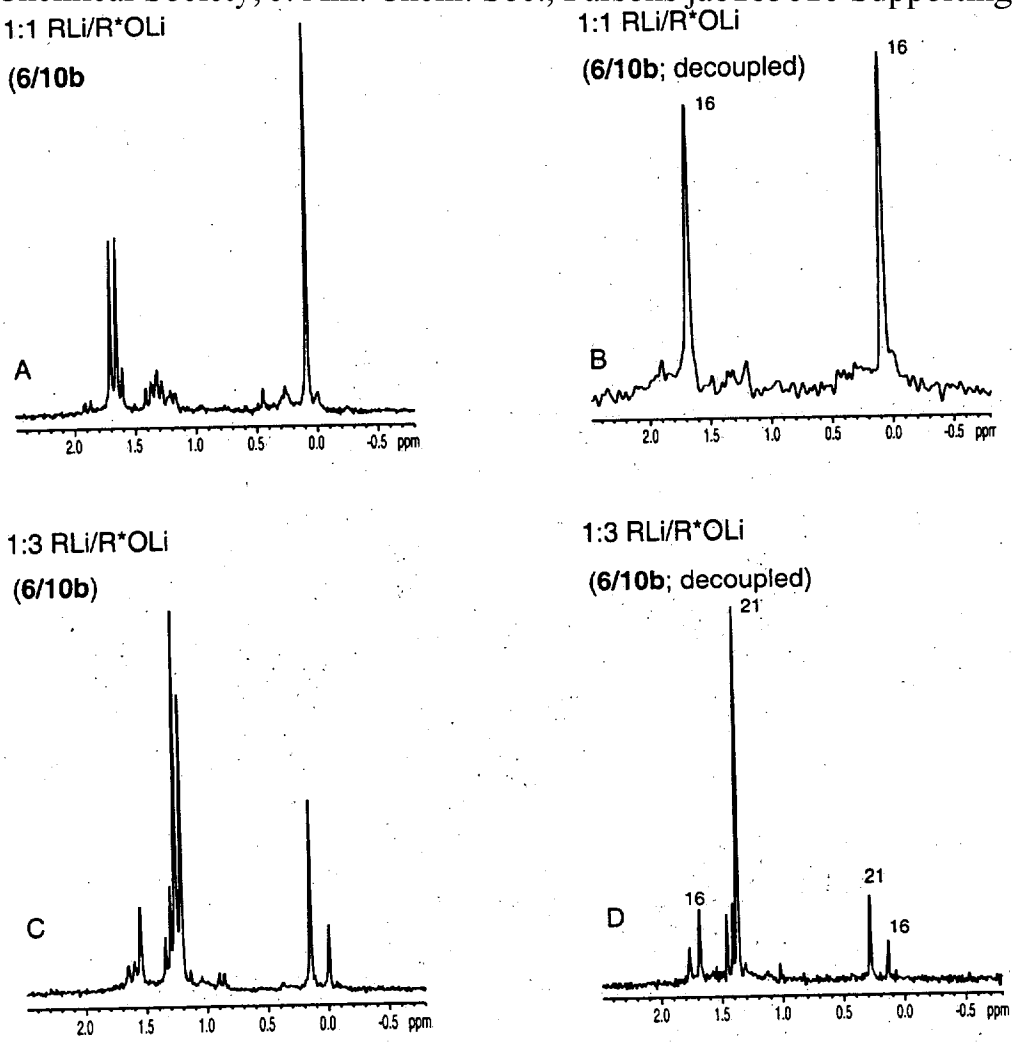

Figure 32. ^{6}Li NMR spectra showing 2:2 RLi/R*OLi (**16**), 1:3 RLi/R*OLi (**21**) mixed tetramers. Spectra were recorded on mixtures of [^{6}Li]LiCPA and [^{6}Li,^{15}N]**10b** (prepared in situ from the alcohol and 1.3 equiv. of [^{6}Li]LiHMDS(*)) in diethyl ether at –95 °C. The total titer of **6** and **10b** is 0.1 M. Spectra B and D were recorded with ^{15}N broad-band decoupling.

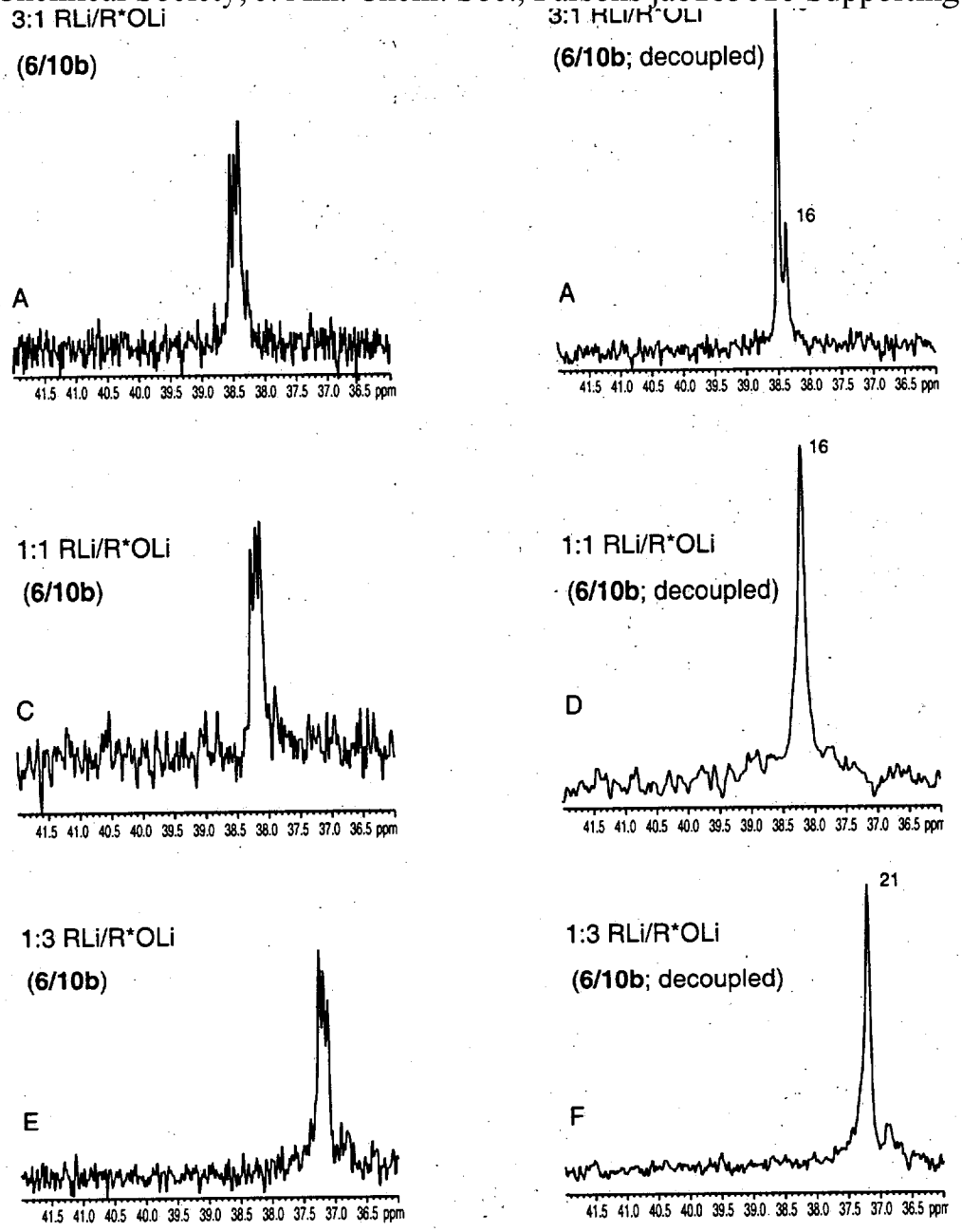

Figure 33. ^{15}N NMR spectra showing 3:1 RLi/R*OLi (**15**), 2:2 RLi/R*OLi (**16**) and 1:3 RLi/R*OLi (**21**) mixed tetramers. Spectra were recorded on mixtures of [^{6}Li]LiCPAand [^{6}Li,^{15}N]**10b** (prepared in situ from the alcohol and 1.3 equiv. of [^{6}Li]LiHMDS(*)) in diethyl ether at –95 °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.

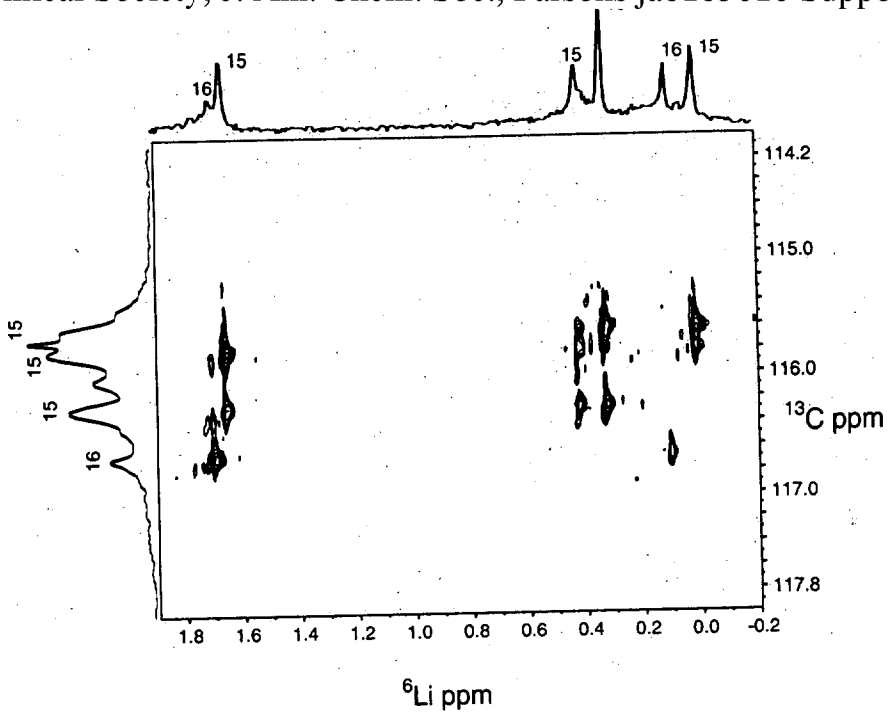

Figure 34. Spectrum of 3:1 RLi/R*OLi showing mixed tetramers **15** and **16**. Spectra were recorded on mixtures of [^{6}Li,^{13}C]LiCPA and [^{6}Li]**10b** (prepared in situ from the alcohol and 1.3 equiv. of [^{6}Li]LiHMDS(*)) in diethyl ether at –95 °C. The total titer of **6** and **10b** is 0.1 M. (A) ^{6}Li,^{13}C-HMQC of 3:1 [^{6}Li,^{13}C]**6**/[^{6}Li]**10b**.

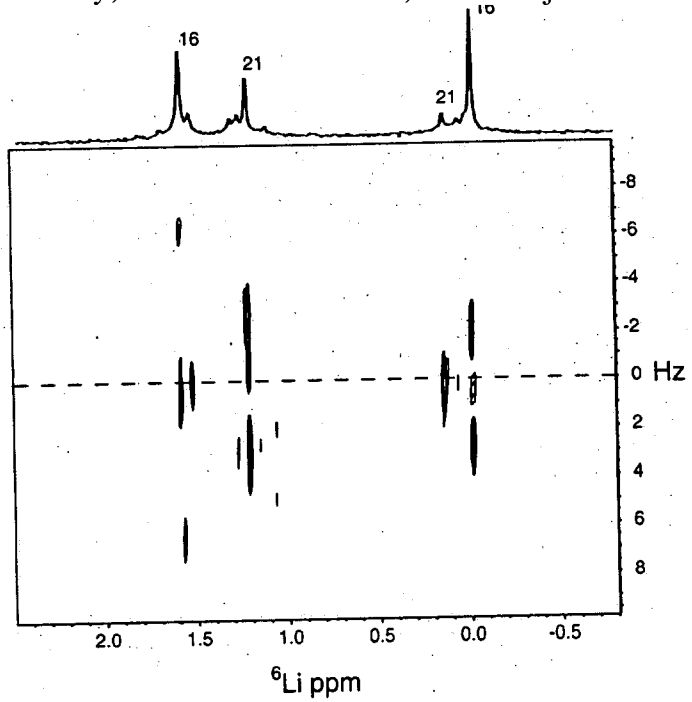

Figure 35. Spectrum of 2:2 RLi/R*OLi showing mixed tetramers **16** and **21**. Spectra were recorded on mixtures of [^{6}Li,^{13}C]LiCPA and [^{6}Li]**10b** (prepared in situ from the alcohol and 1.3 equiv. of [^{6}Li]LiHMDS(*)) in diethyl ether at –95 °C. The total titer of **6** and **10b** is 0.1 M. (A) *J*-resolved spectrum of 2:2 [^{6}Li,^{13}C]**6**/[^{6}Li]**10b**.

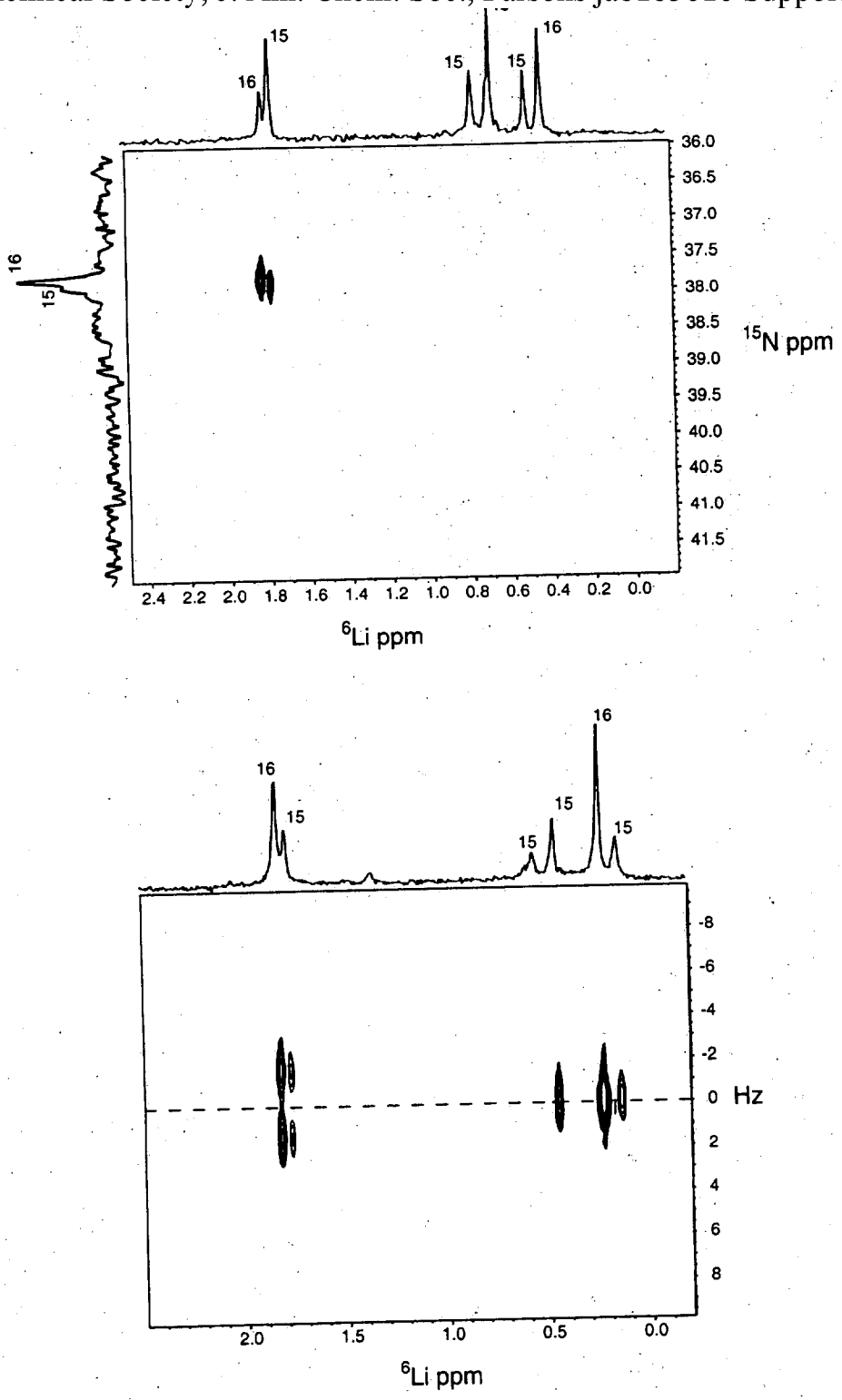

Figure 36. Spectra of 3:1 RLi/R*OLi showing mixed tetramers **15**, **16**, and **21**. Spectra were recorded on mixtures of [^{6}Li]LiCPA and [^{6}Li,^{15}N]**10b** (prepared in situ from the alcohol and 1.3 equiv. of [^{6}Li]LiHMDS(*)) in diethyl ether at –95 °C. The total titer of **6** and **10b** is 0.1 M. (A) ^{6}Li,^{15}N -HMQC of 1:1 [^{6}Li,^{15}N]**6**/[^{6}Li]**10b**; (B) ^{6}Li *J*-resolved spectrum of 1:1 [^{6}Li,^{15}N]**6**/[^{6}Li]**10b**.

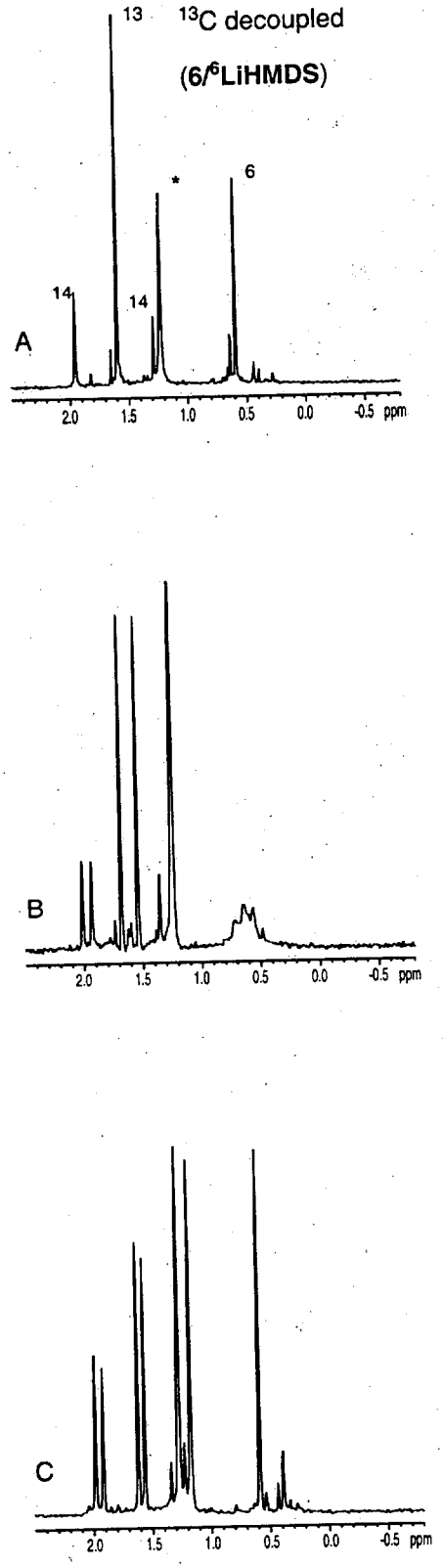

Figure 37. ^{6}Li NMR spectra showing a 1:2 mix of **6** and [^{6}Li]LiHMDS(*)). (A) [^{6}Li,^{13}C]LiCPA and [^{6}Li]LiHMDS(*)) with ^{13}C broad band decoupling; (B) [^{6}Li,^{13}C]LiCPA and [^{6}Li]LiHMDS(*)); (C) [^{6}Li]LiCPA and [^{6}Li,^{15}N]LiHMDS(*)). All spectra were recorded in DMEA at –100 °C.

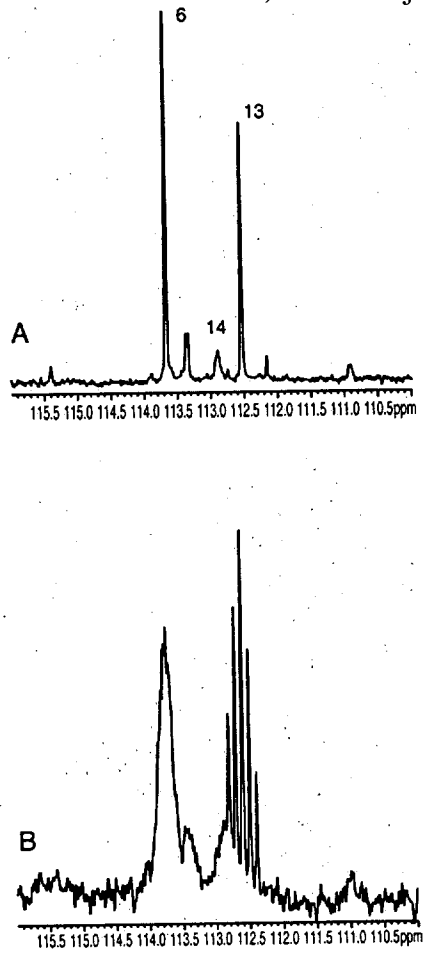

Figure 38. ^{13}C NMR spectra showing a 1:1 mix of **6** and [^{6}Li]LiHMDS(*)). (A) [^{6}Li,^{13}C]LiCPA and [^{6}Li]LiHMDS(*)) with ^{6}Li decoupling; (B) [^{6}Li,^{13}C]LiCPA and [^{6}Li]LiHMDS(*)). All spectra were in DMEA at –100 °C.

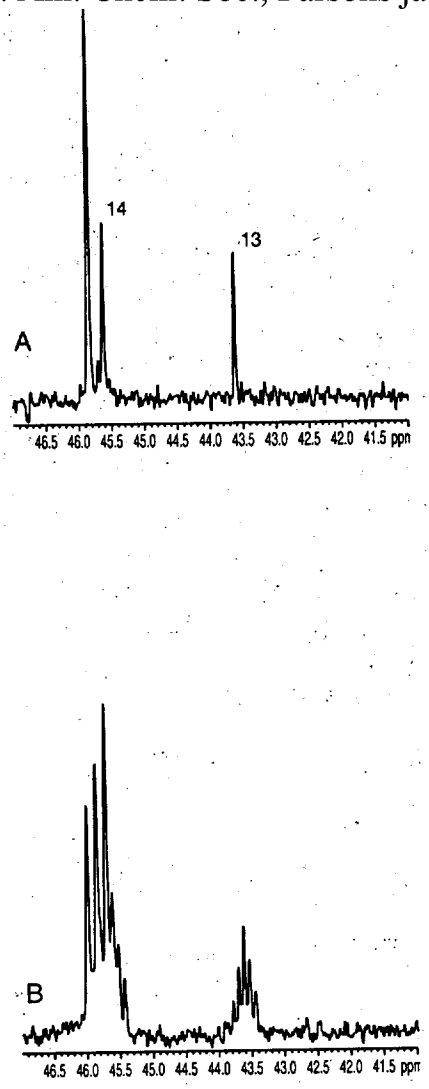

Figure 39. ^{15}N NMR spectra showing a 1:2 mix of **6** and [^{6}Li,^{15}N]LiHMDS(*)). (A) [^{6}Li]LiCPA and [^{6}Li,^{15}N]LiHMDS(*)) with ^{6}Li decoupling (B) [^{6}Li]LiCPA and [^{6}Li,^{15}N]LiHMDS(*)). All spectra were recorded in DMEA at –100 °C.

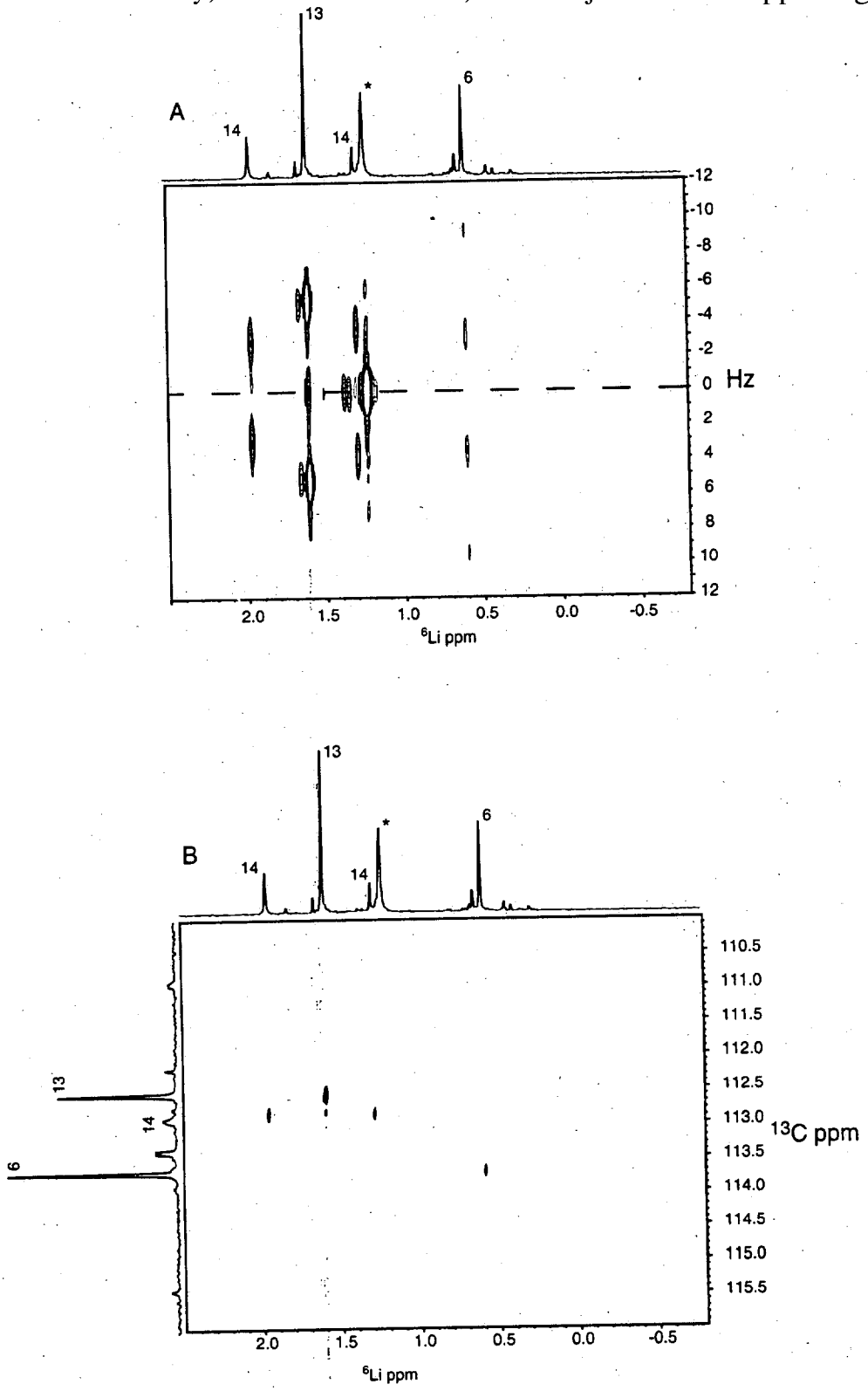

Figure 40. Spectra of a 1:2 mix of **6** and [^{6}Li]LiHMDS(*)). Spectra were recorded on mixtures of [^{6}Li,^{13}C]LiCPA and [^{6}Li]LiHMDS(*)) in DMEA at –100 °C. (A) ^{6}Li *J*-resolved spectrum of 1:2 [^{6}Li,^{13}C]LiCPA/[^{6}Li]LiHMDS(*)). (B) ^{6}Li,^{13}C-HMQC of 1:2 [^{6}Li,^{13}C]LiCPA/[^{6}Li]LiHMDS(*)).

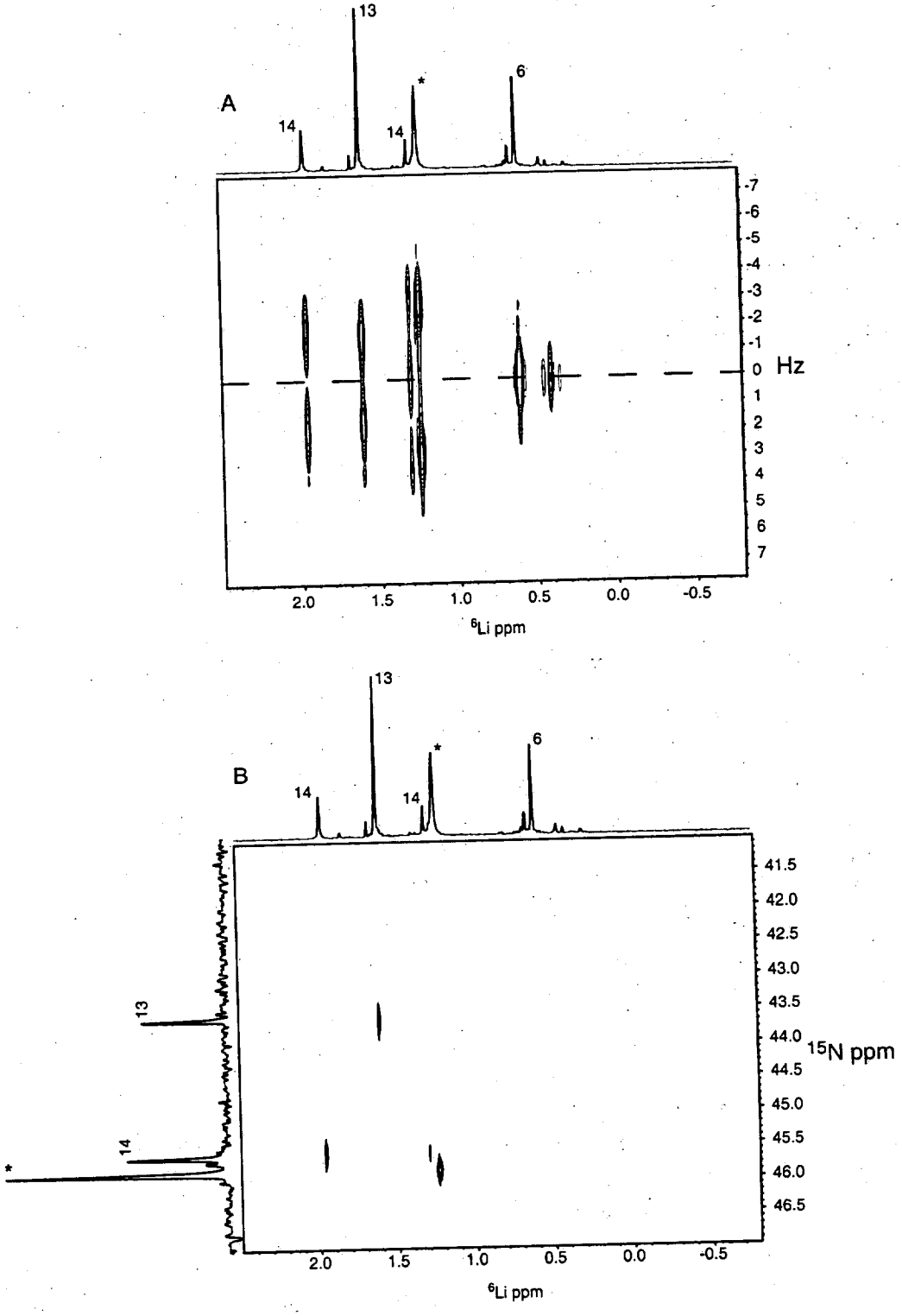

Figure 41. Spectra of a 1:2 mix of **6** and [^{6}Li,^{15}N]LiHMDS(*)). Spectra were recorded on mixtures of [^{6}Li]LiCPA and [^{6}Li,^{15}N]LiHMDS(*)) in DMEA at –100 °C. (A) *J*-resolved spectrum of 1:2 [^{6}Li]LiCPA/[^{6}Li,^{15}N]LiHMDS(*)). (B) ^{6}Li,^{15}N-HMQC of 1:2 [^{6}Li]LiCPA/[^{6}Li,^{15}N]LiHMDS(*)).