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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_2NLi in 3:2 pentane:toluene at varying temperatures.

II. ^{15}N NMR spectra of 0.1 M [^{6}Li,^{15}N]*i*-Pr_2NLi 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_2NLi 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_2NLi 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_2NLi 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_2NLi 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_2NLi 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_2NLi 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_2NLi 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_2NLi 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_2NLi in 3:2 pentane:toluene with 0.25 equiv of THF.

XII. ^{6}Li and ^{15}N NMR spectra of 0.1 M [^{6}Li,^{15}N]*i*-Pr_2NLi 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}Li,^{15}N]*i*-Pr_2NLi in 3:2 pentane:toluene with 0.5 equiv of THF.

XIV. ^{6}Li,^{15}N-heteronuclear multiple quantum correlation (HMQC) spectrum of 0.1 M [^{6}Li,^{15}N]*i*-Pr_2NLi in 3:2 pentane:toluene with 0.75 equiv of THF.

XV. ^{6}Li NMR spectra of 0.1 M [^{6}Li,^{15}N]*i*-Pr_2NLi in 3:2 pentane:toluene with varying amounts of THF.

XVI. ^{6}Li and ^{15}N NMR spectra of 0.1 M [^{6}Li,^{15}N]*i*-Pr_2NLi 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}Li,^{15}N]*i*-Pr_2NLi in 3:2 pentane:toluene with 0.25 equiv of diethyl ether.

XVIII. ^{6}Li NMR spectra of 0.1 M [^{6}Li,^{15}N]*i*-Pr_2NLi in 3:2 pentane:toluene with varying amounts of diethyl ether.

XIX. ^{6}Li NMR spectra of 0.1 M [^{6}Li,^{15}N]*i*-Pr_2NLi in 3:2 pentane:toluene with varying amounts of diisopropylamine.

XX. ^{6}Li NMR spectra of 0.1 M [^{6}Li]*i*-Pr_2NLi in 3:2 pentane:toluene with varying amounts of diisopropylamine.

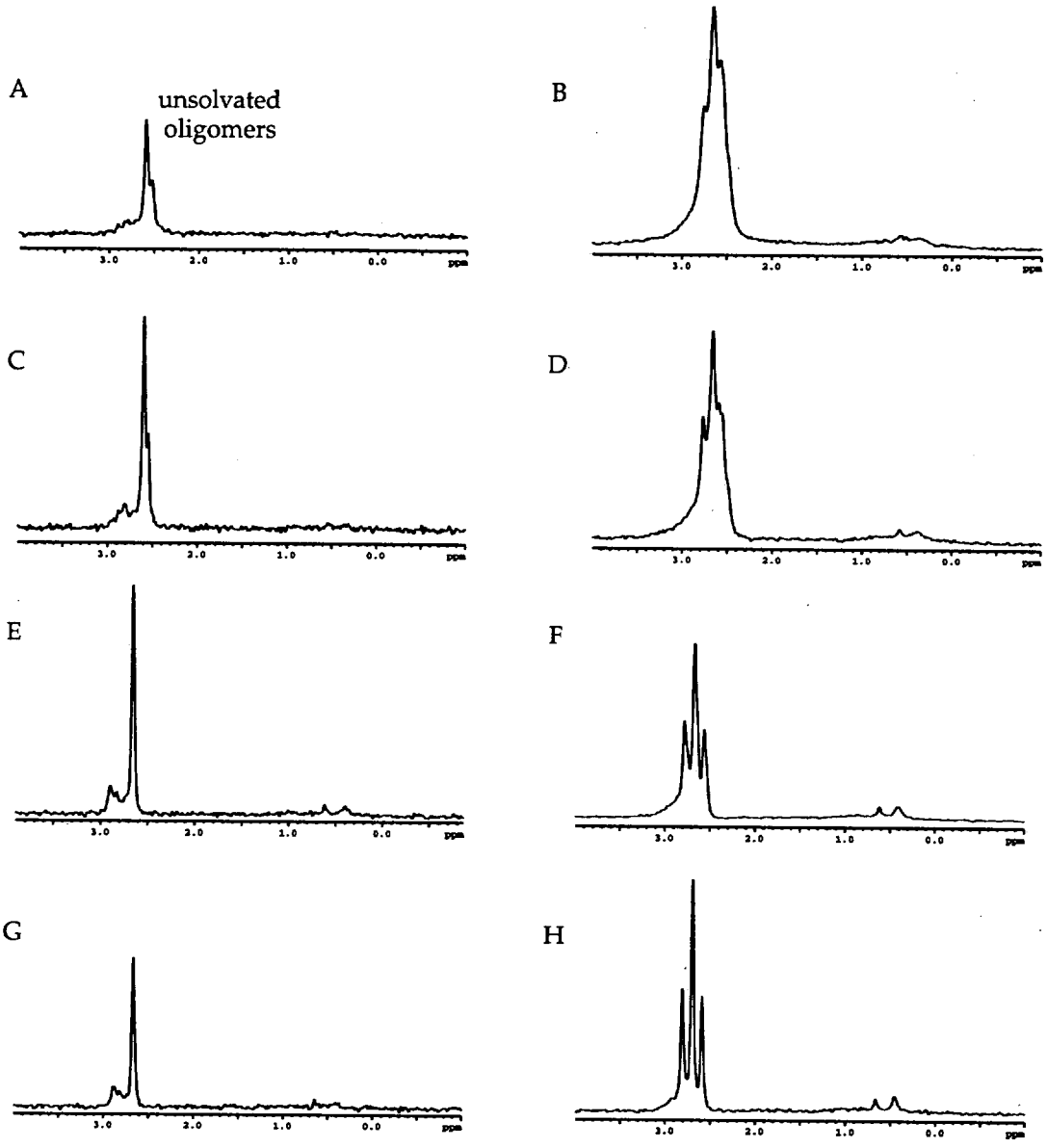

Figure I. ^{6}Li NMR spectra of 0.1 M [^{6}Li,^{15}N]*i*-Pr_2NLi in 3:2 pentane:toluene: (A) $^{6}Li\{^{15}N\}$ spectrum at -135 °C; (B) ^{6}Li spectrum at -135 °C; (C) $^{6}Li\{^{15}N\}$ spectrum at -127 °C; (D) ^{6}Li spectrum at -127 °C; (E) $^{6}Li\{^{15}N\}$ spectrum at -110 °C; (F) ^{6}Li spectrum at -110 °C; (G) $^{6}Li\{^{15}N\}$ spectrum at -100 °C; (H) ^{6}Li spectrum at -100 °C.

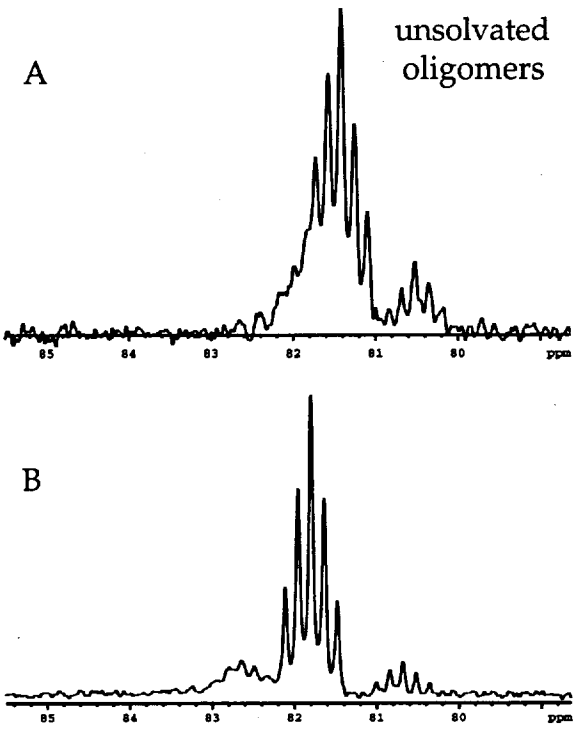

Figure II. ^{15}N NMR spectra of 0.1 M [^{6}Li,^{15}N]*i*-Pr_2NLi in 3:2 pentane:toluene: (A)$^{15}N\{^{1}H\}$ spectrum at -127 °C; (B) $^{15}N\{^{1}H\}$ spectrum at -100 °C.

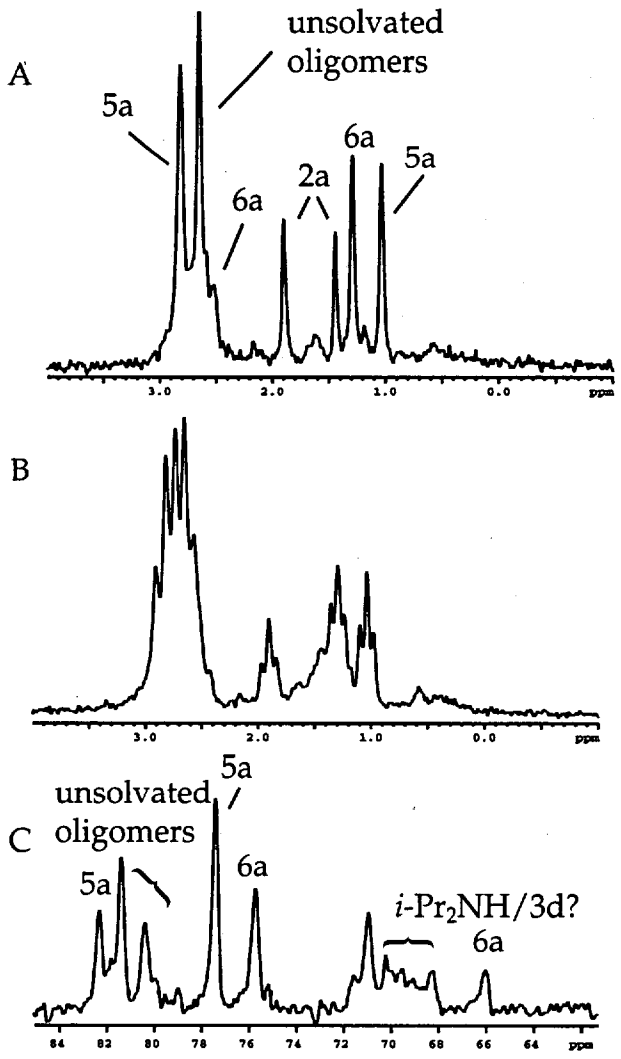

Figure III. ^{6}Li and ^{15}N NMR spectra of 0.1 M [^{6}Li,^{15}N]*i*-Pr_2NLi in 3:2 pentane:toluene with 0.25 equiv of oxetane at -135 °C: (A) $^{6}Li\{^{15}N\}$ spectrum; (B) ^{6}Li spectrum; (C) $^{15}N\{^{1}H, ^{6}Li\}$ spectrum.

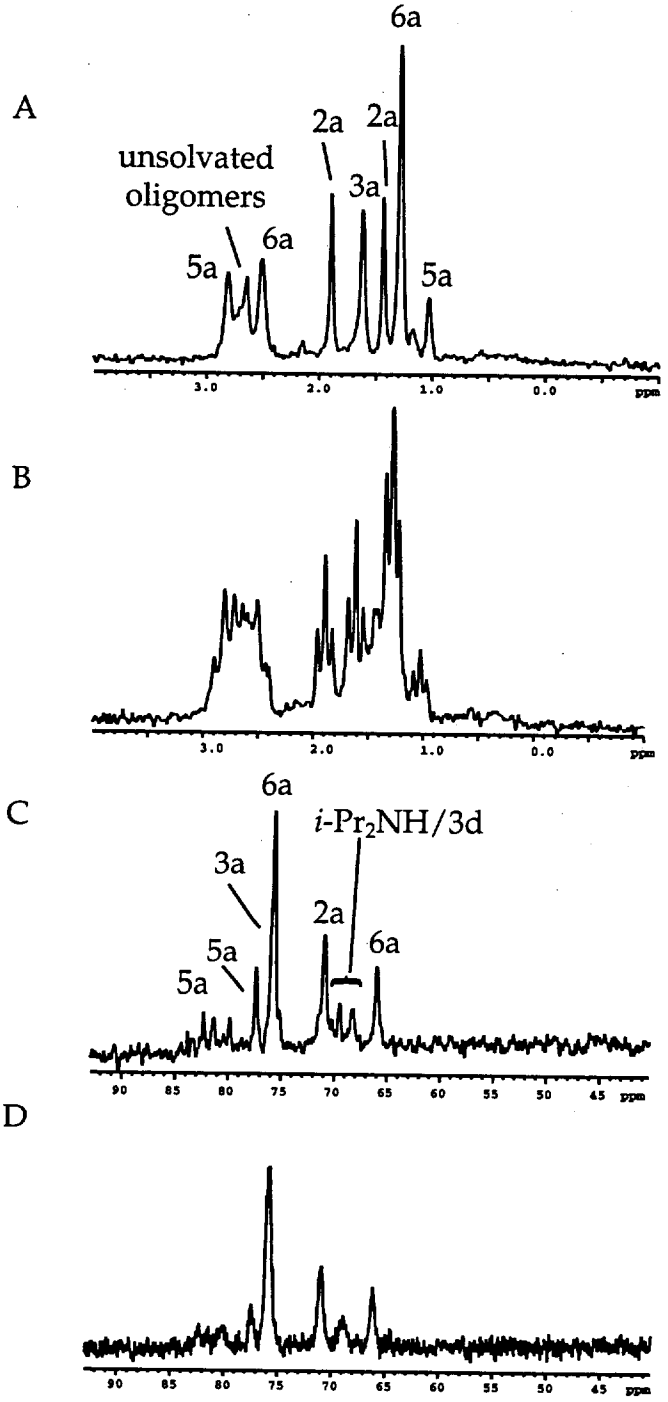

Figure IV. ^{6}Li and ^{15}N NMR spectra of 0.1 M [^{6}Li,^{15}N]*i*-Pr_2NLi in 3:2 pentane:toluene with 0.5 equiv of oxetane at -135 °C: (A) $^{6}Li\{^{15}N\}$ spectrum; (B) ^{6}Li spectrum; (C) $^{15}N\{^{1}H, ^{6}Li\}$ spectrum; (D) $^{15}N\{^{1}H\}$ spectrum.

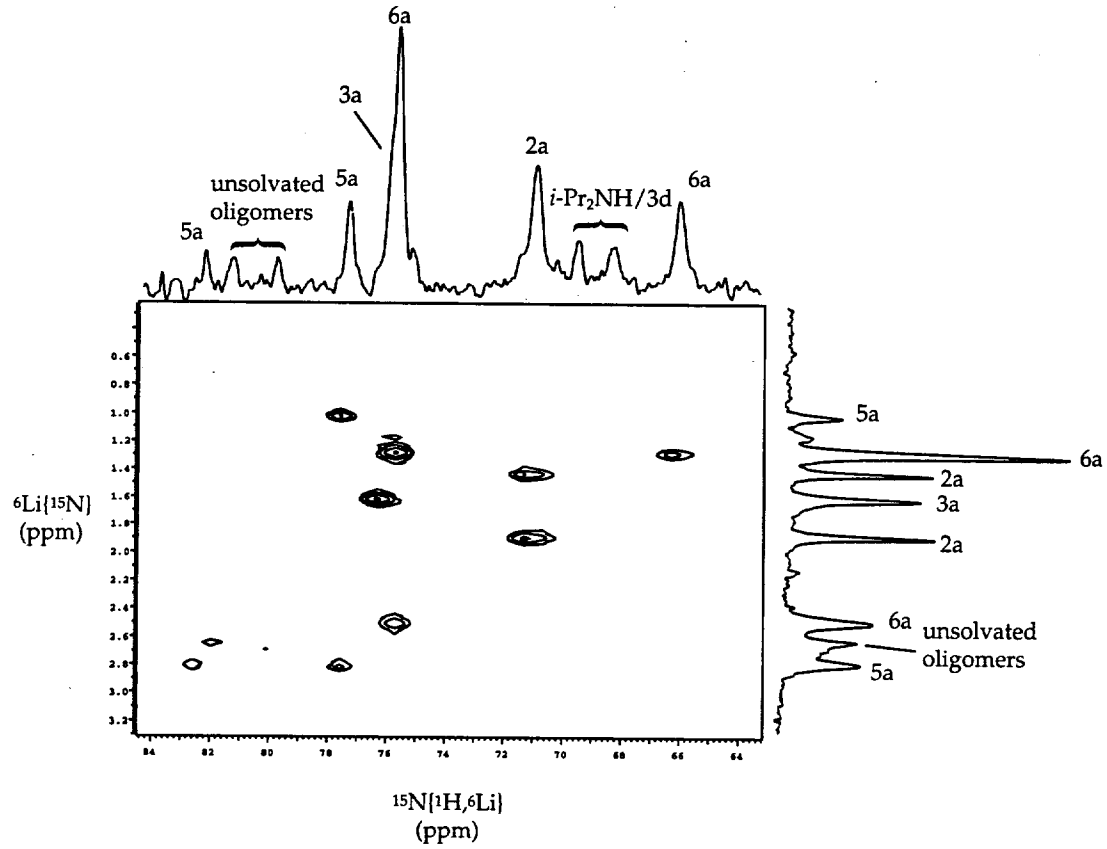

Figure V. $^{6}Li,^{15}N$-heteronuclear multiple quantum correlation (HMQC) spectrum of 0.1 M [$^{6}Li,^{15}N$]*i*-Pr_2NLi in 3:2 pentane:toluene with 0.5 equiv of oxetane at -135 °C.

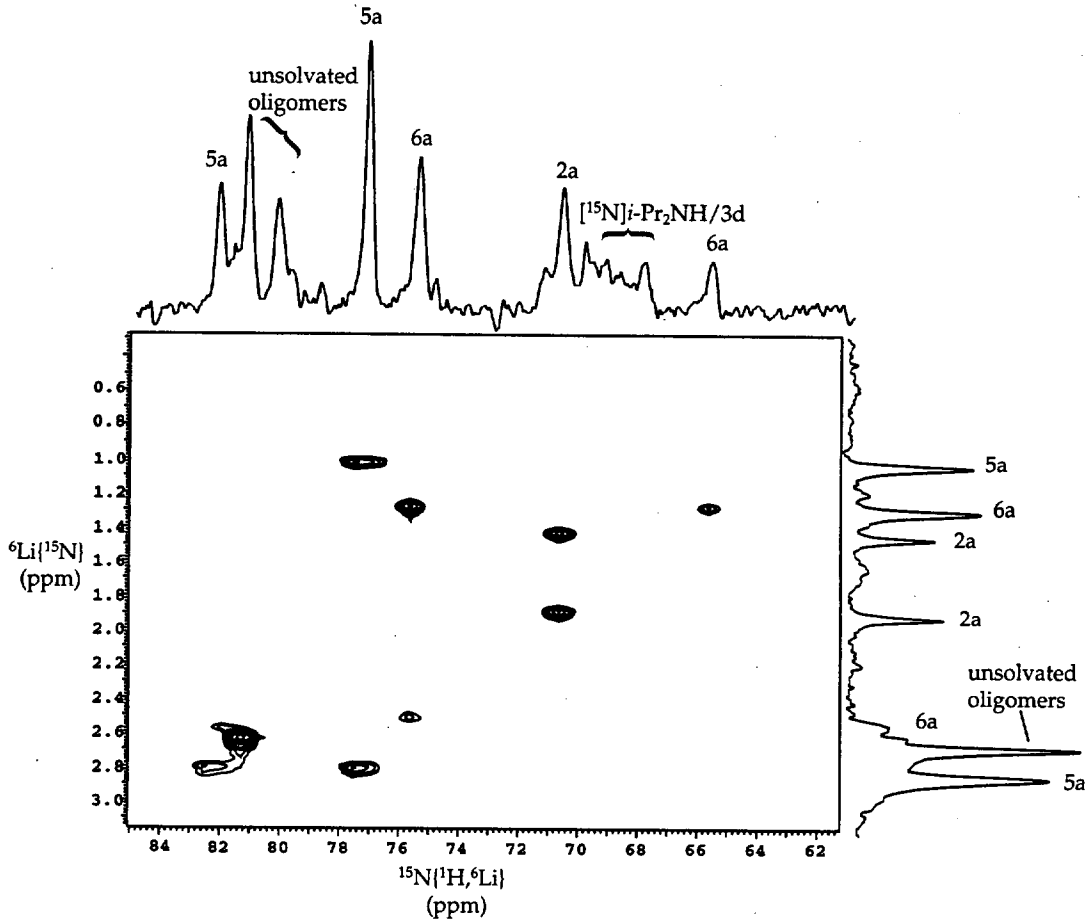

Figure VI. $^{6}Li,^{15}N$-heteronuclear multiple quantum correlation (HMQC) spectrum of 0.1 M [$^{6}Li,^{15}N$]LDA in 3:2 pentane:toluene with 0.25 equiv of oxetane at -135 °C.

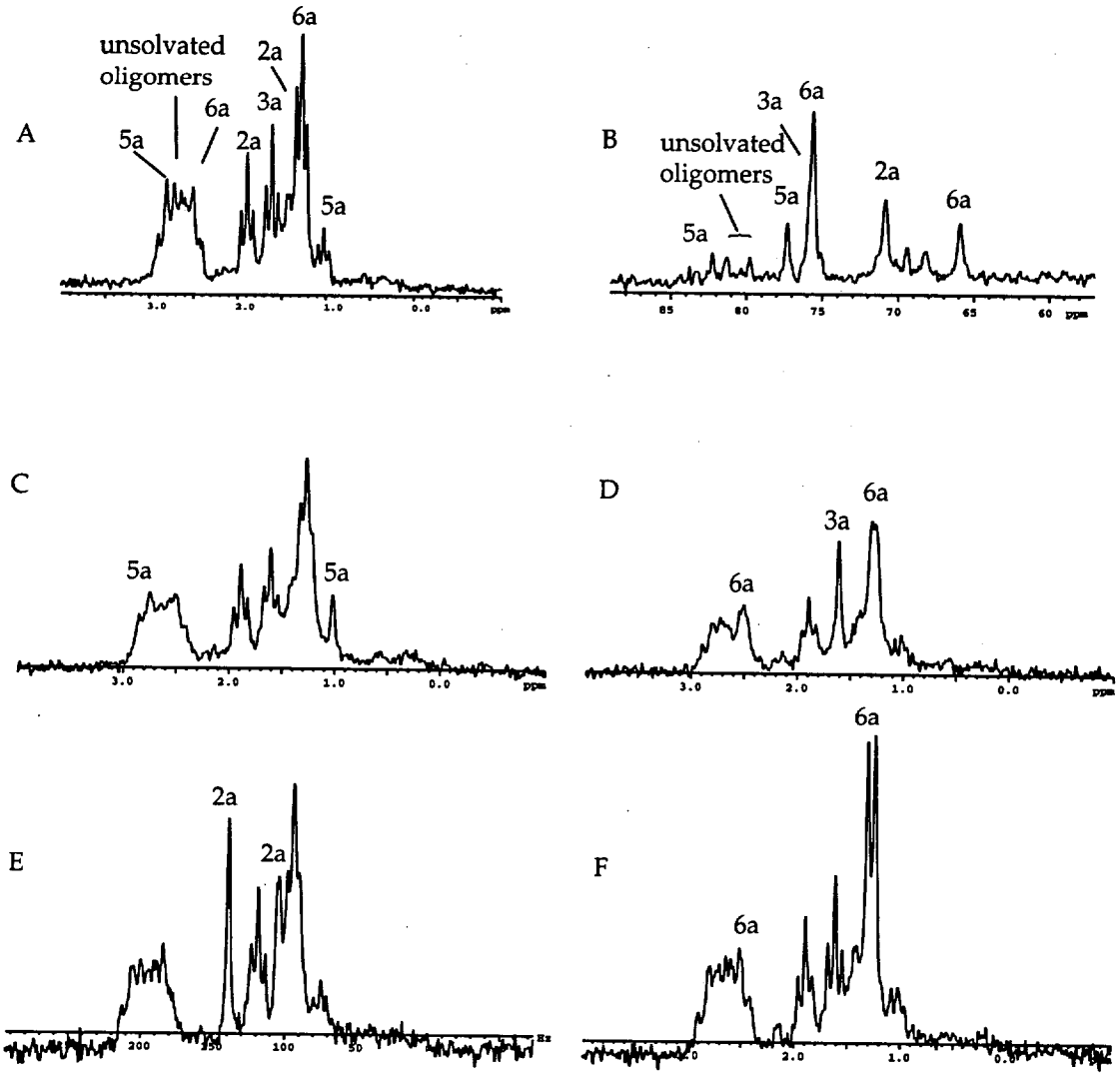

Figure VII. ^{6}Li and ^{15}N NMR spectra of 0.1 M [^{6}Li,^{15}N]*i*-Pr_2NLi in 3:2 pentane:toluene with 0.5 equiv of oxetane at -135 °C: (A) ^{6}Li spectrum; (B) $^{15}N\{^{1}H, ^{6}Li\}$ spectrum; (C) ^{6}Li spectrum, ^{15}N single frequency decoupling at 77.4 ppm; (D) ^{6}Li spectrum, ^{15}N single 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.

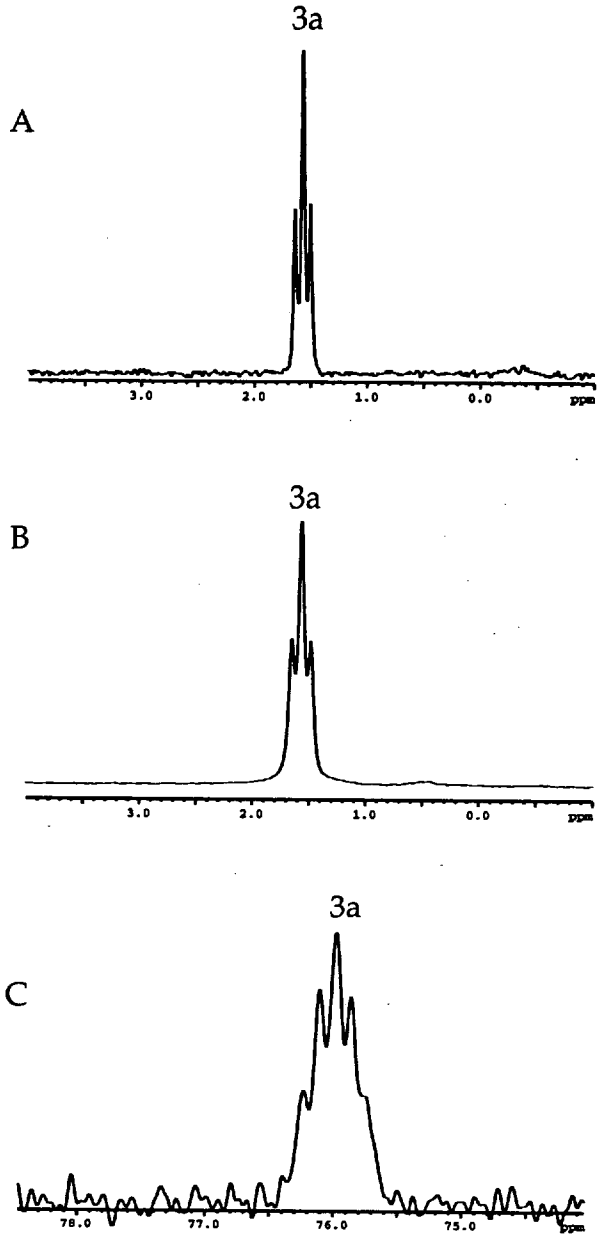

Figure VIII. ^{6}Li and ^{15}N NMR spectra of 0.1 M [^{6}Li,^{15}N]*i*-Pr_2NLi in 3:2 pentane:toluene: (A) ^{6}Li spectrum with 10 equiv oxetane at -120 °C; (B) ^{6}Li spectrum with 1.25 equiv oxetane at -135 °C; (C) $^{15}N\{^{1}H\}$ spectrum with 1.25 equiv oxetane at -135 °C.

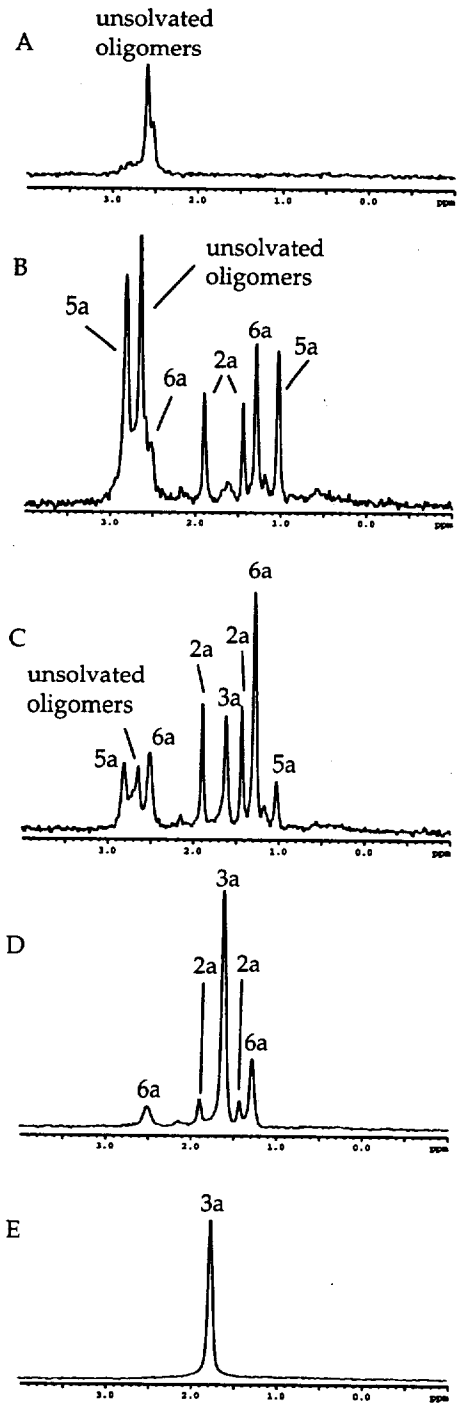

Figure IX. $^{6}Li\{^{15}N\}$ NMR spectra of 0.1 M [^{6}Li,^{15}N]LDA in 3:2 pentane:toluene at -135 °C with: (A) no added ligand; (B) 0.25 equiv oxetane; (C) 0.5 equiv oxetane; (D) 0.75 equiv oxetane; (E) 1.0 equiv oxetane.

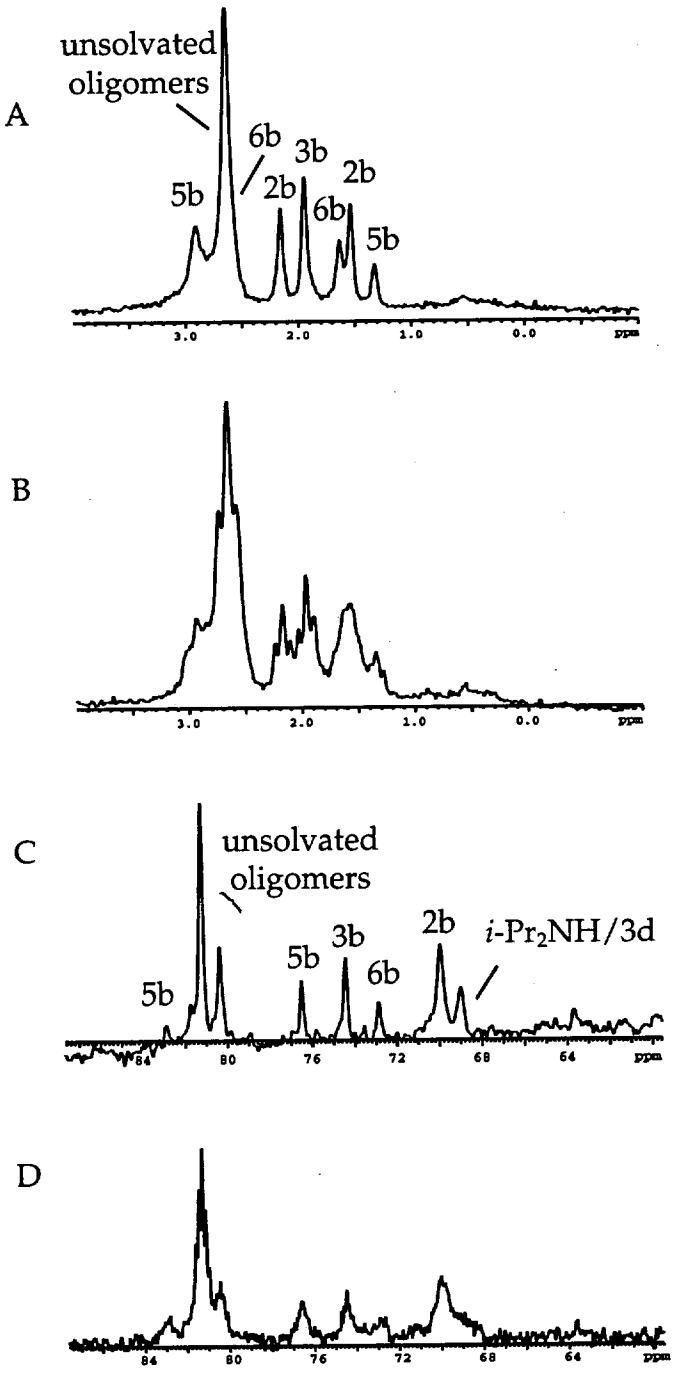

Figure X. ^{6}Li and ^{15}N NMR spectra of 0.1 M [^{6}Li,^{15}N]*i*-Pr_2NLi in 3:2 pentane:toluene with 0.25 equiv of THF at -135 °C: (A) $^{6}Li\{^{15}N\}$ spectrum; (B) ^{6}Li spectrum; (C) $^{15}N\{^{1}H, ^{6}Li\}$ spectrum; (D) $^{15}N\{^{1}H\}$ spectrum.

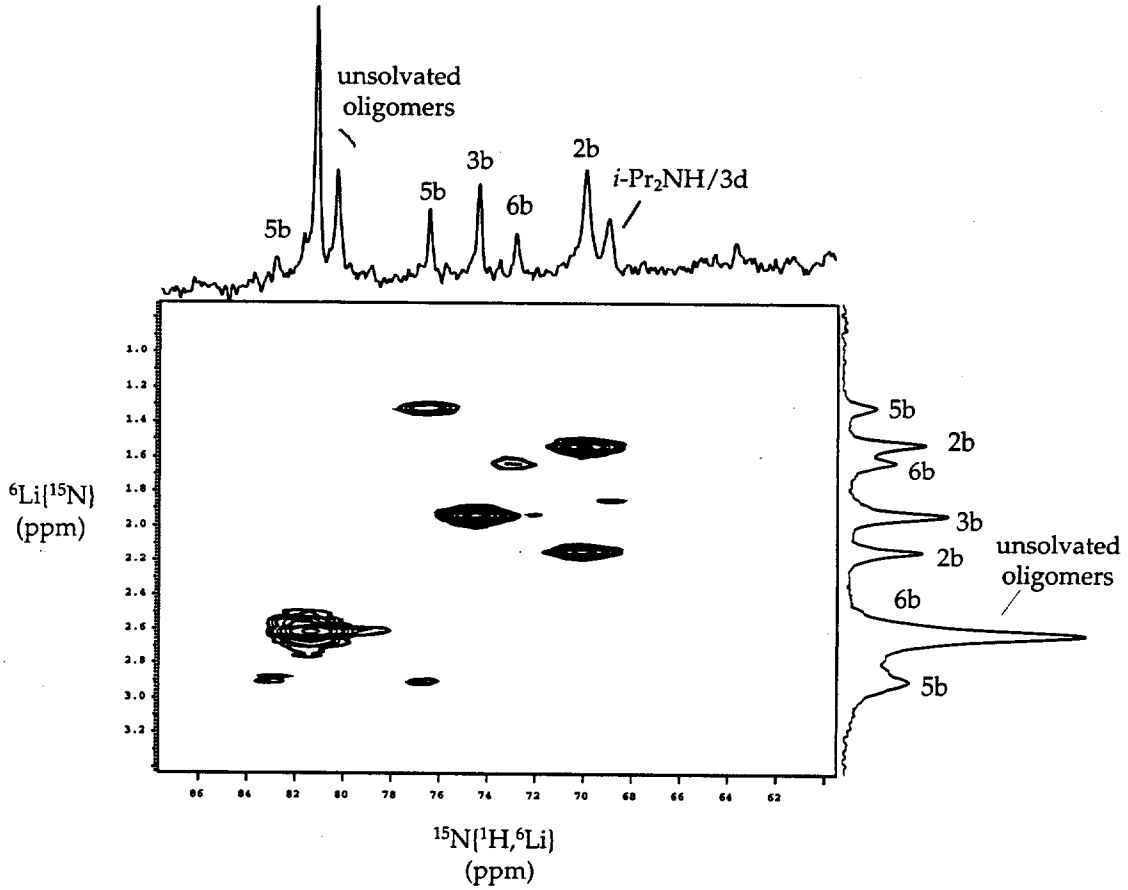

Figure XI. $^{6}Li,^{15}N$-heteronuclear multiple quantum correlation (HMQC) spectrum of 0.1 M [$^{6}Li,^{15}N$]*i*-Pr_2NLi in 3:2 pentane:toluene with 0.25 equiv of THF at -135 °C.

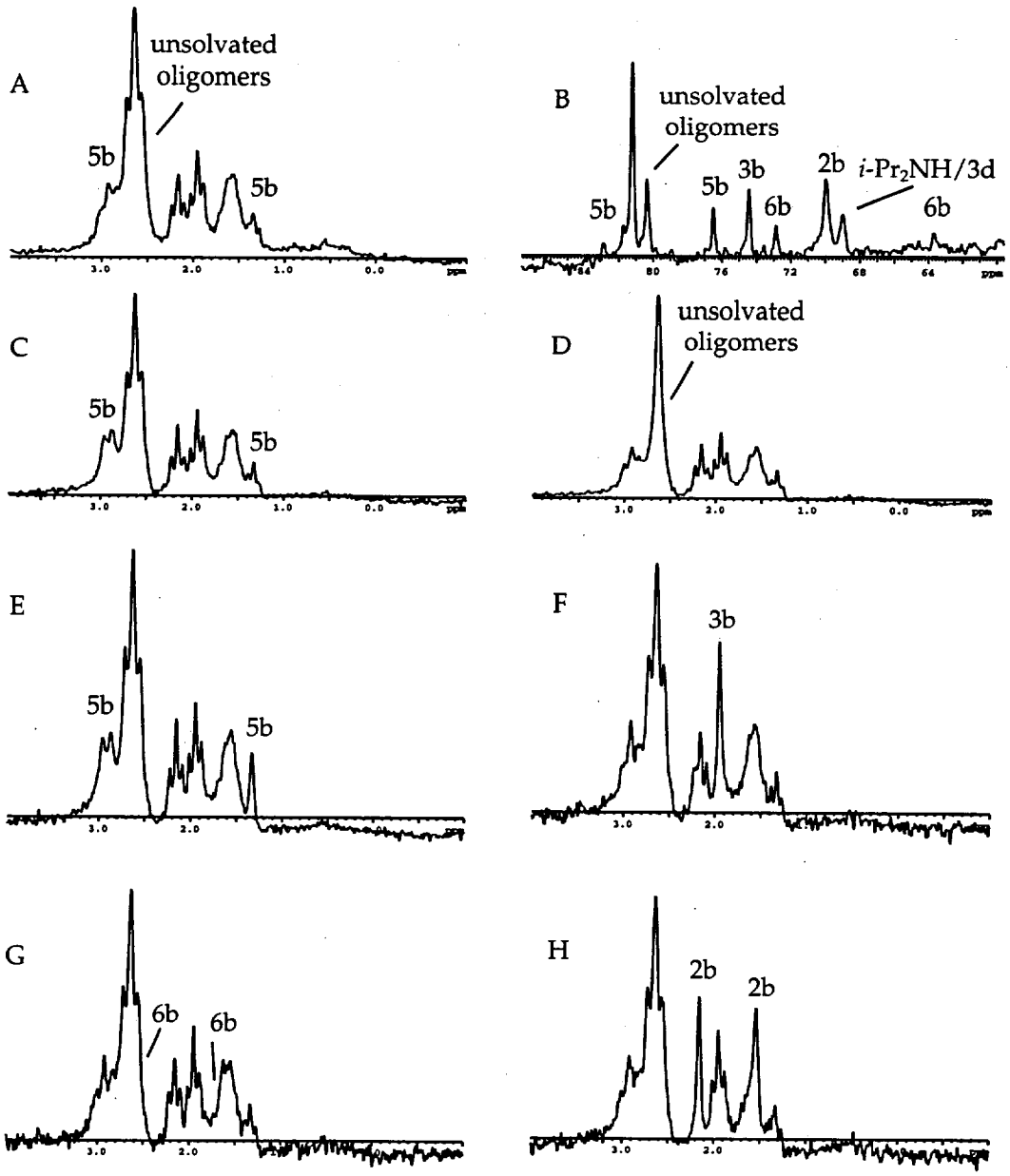

Figure XII. ^{6}Li and ^{15}N NMR spectra of 0.1 M [^{6}Li,^{15}N]*i*-Pr_2NLi in 3:2 pentane:toluene with 0.25 equiv of THF at -135 °C: (A) ^{6}Li spectrum; (B) $^{15}N\{^{1}H, ^{6}Li\}$ spectrum; (C) ^{6}Li spectrum, ^{15}N single frequency decoupling at 83.0 ppm; (D) ^{6}Li spectrum, ^{15}N single 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.

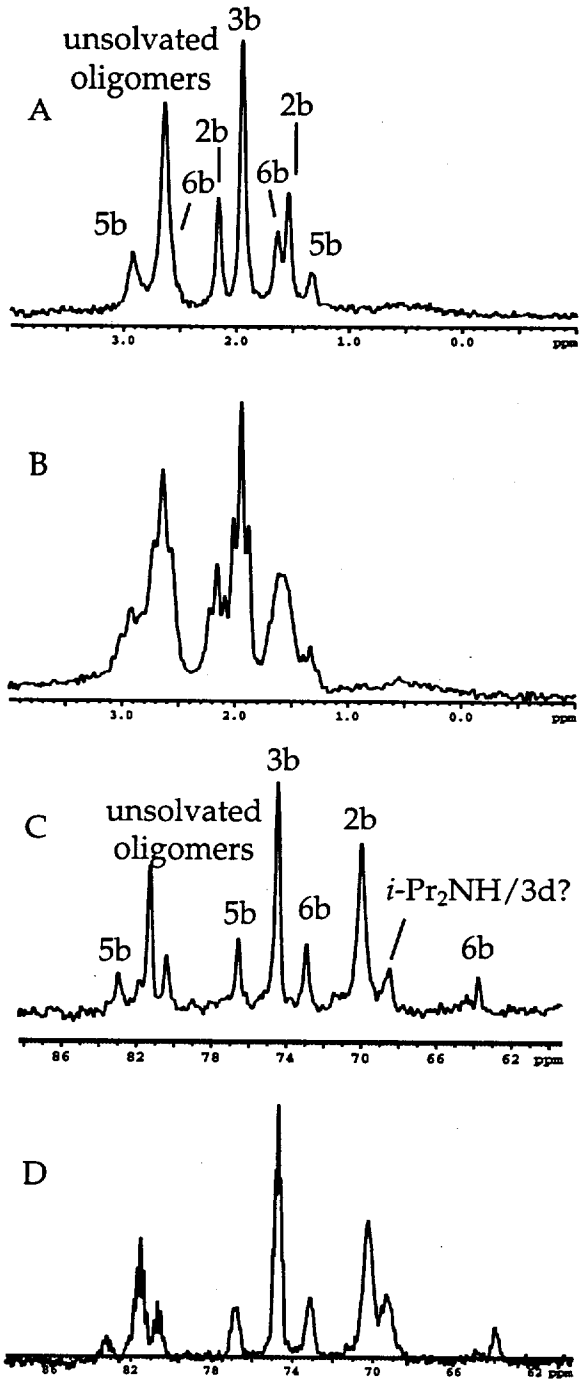

Figure XIII. ^{6}Li and ^{15}N NMR spectra of 0.1 M [^{6}Li,^{15}N]*i*-Pr_2NLi in 3:2 pentane:toluene with 0.5 equiv of THF at -135 °C: (A) $^{6}Li\{^{15}N\}$ spectrum; (B) ^{6}Li spectrum; (C) $^{15}N\{^{1}H, ^{6}Li\}$ spectrum; (D) $^{15}N\{^{1}H\}$ spectrum.

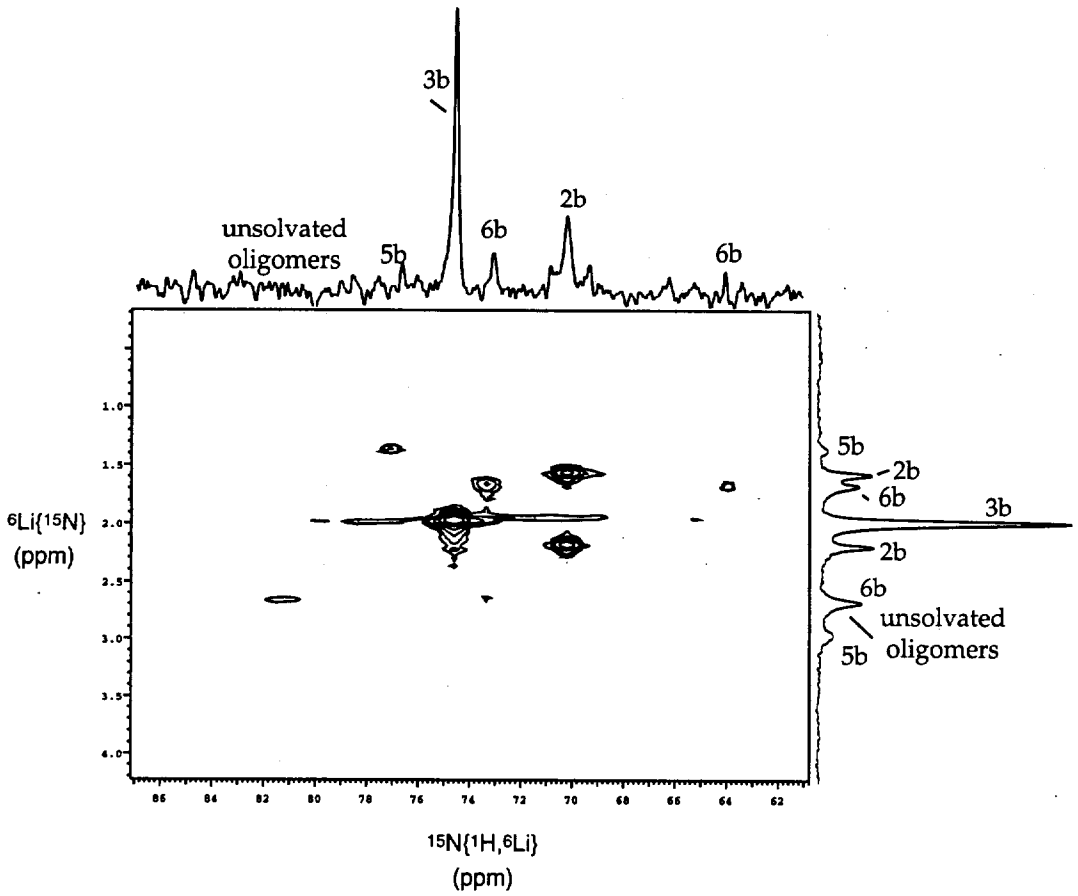

Figure XIV. $^{6}Li,^{15}N$-heteronuclear multiple quantum correlation (HMQC) spectrum of 0.1 M [$^{6}Li,^{15}N$]*i*-Pr_2NLi in 3:2 pentane:toluene with 0.75 equiv of THF at -135 °C.

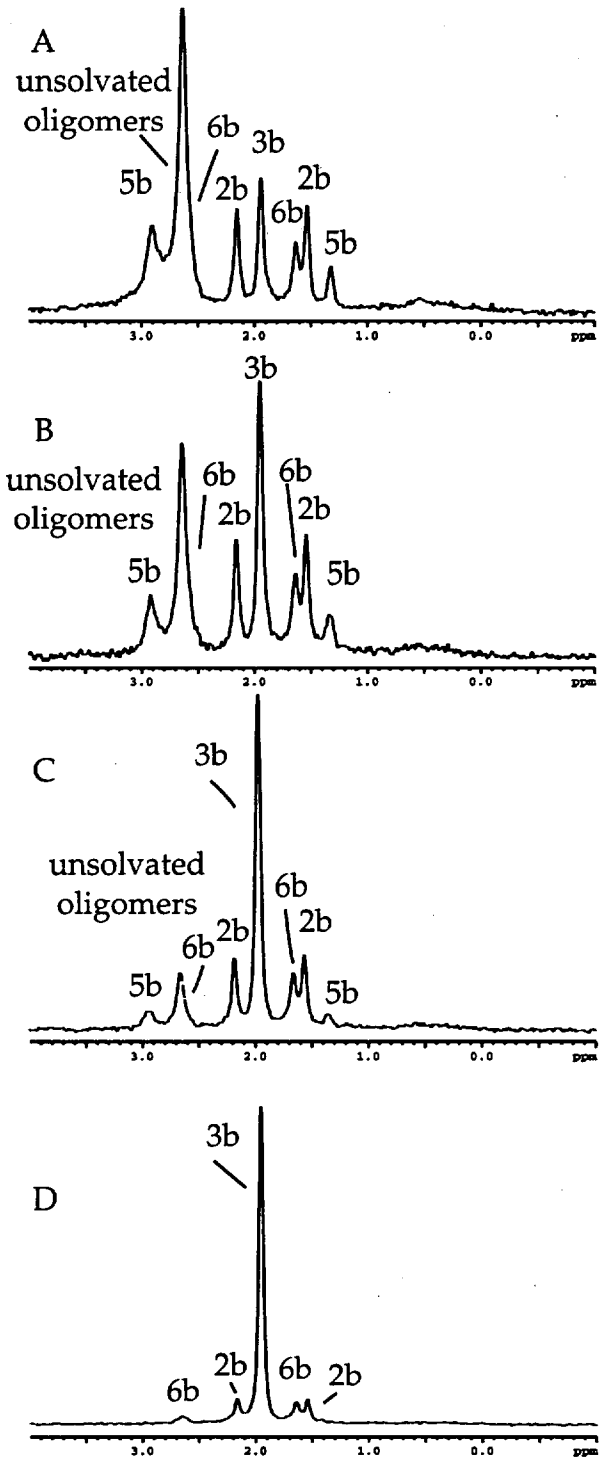

Figure XV. ^{6}Li and ^{15}N NMR spectra of 0.1 M [^{6}Li,^{15}N]*i*-Pr_2NLi in 3:2 pentane:toluene at -135 °C with: (A) 0.25 equiv THF; (B) 0.5 equiv THF; (C) 0.75 equiv THF; (D) 1.0 equiv THF.

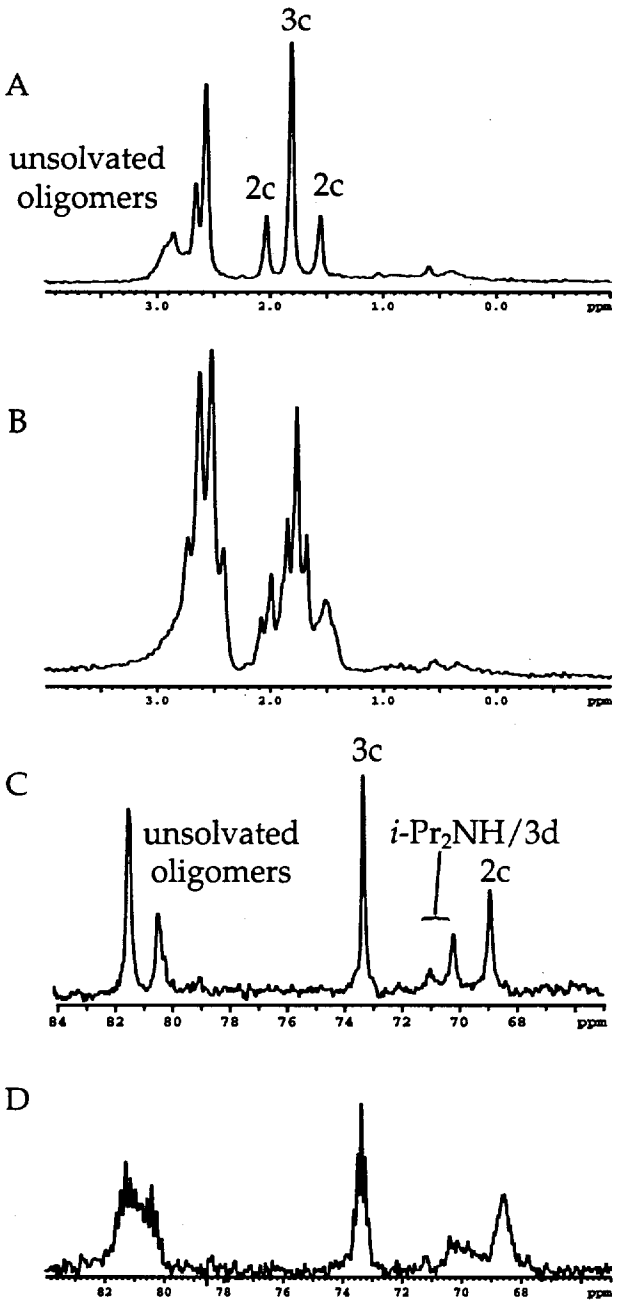

Figure XVI. ^{6}Li and ^{15}N NMR spectra of 0.1 M [^{6}Li,^{15}N]*i*-Pr_2NLi in 3:2 pentane:toluene with 0.25 equiv of diethyl ether at -127 °C: (A) $^{6}Li\{^{15}N\}$ spectrum; (B) ^{6}Li spectrum; (C) $^{15}N\{^{1}H, ^{6}Li\}$ spectrum; (D) $^{15}N\{^{1}H\}$ spectrum.

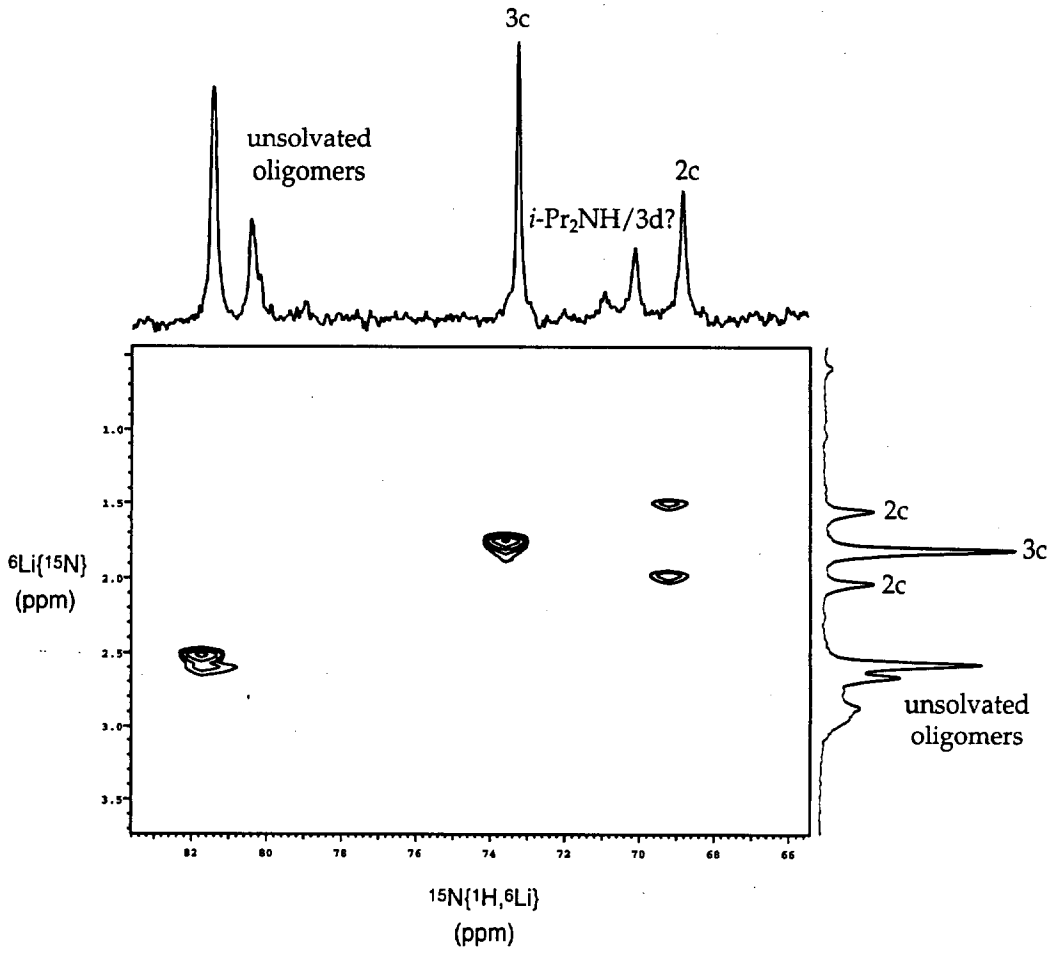

Figure XVII. $^{6}Li,^{15}N$-heteronuclear multiple quantum correlation (HMQC) spectrum of 0.1 M [$^{6}Li,^{15}N$]*i*-Pr_2NLi in 3:2 pentane:toluene with 0.25 equiv of diethyl ether at -127 °C.

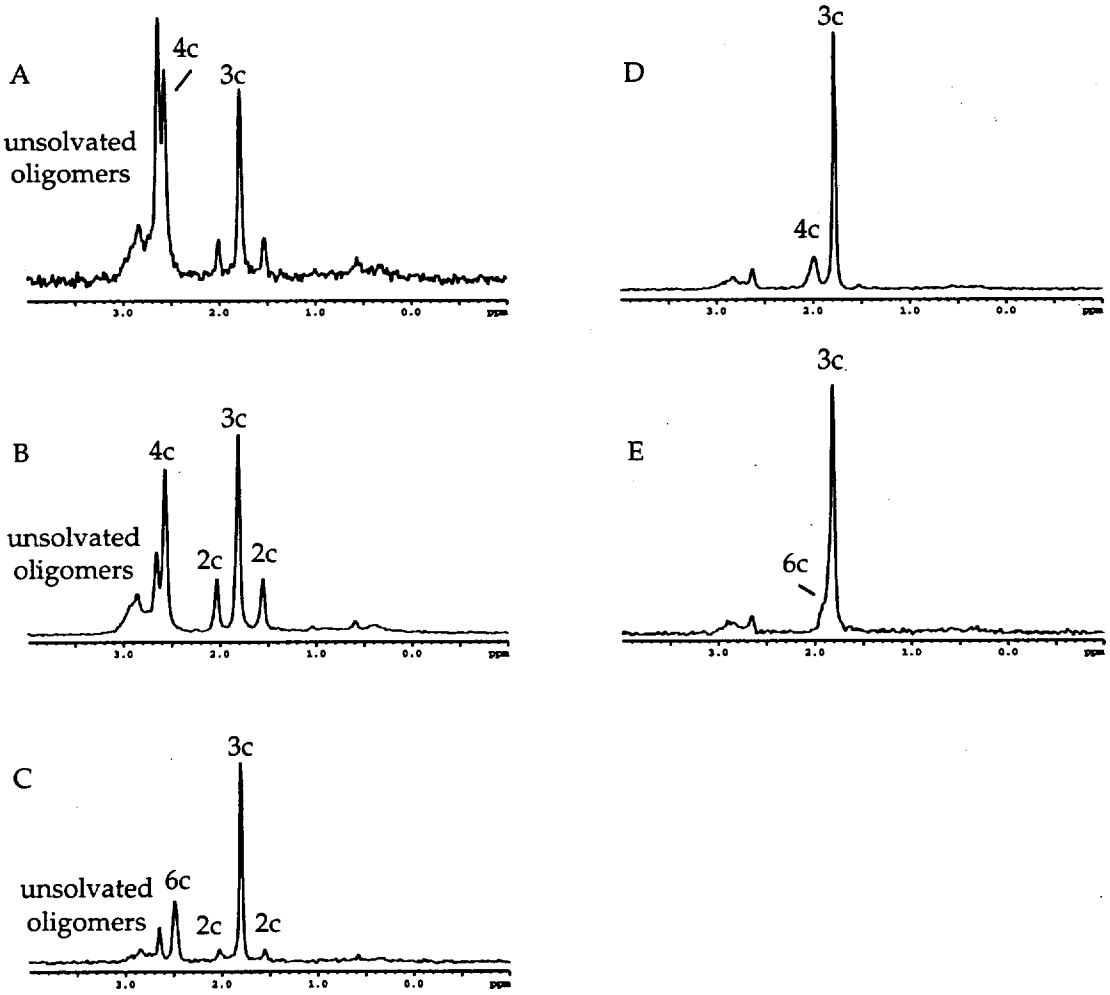

Figure XVIII. ^{6}Li NMR spectra of 0.1 M [^{6}Li,^{15}N]*i*-Pr_2NLi in 3:2 pentane:toluene at -127 °C with: (A) 0.1 equiv diethyl ether; (B) 0.25 equiv diethyl ether; (C) 0.5 equiv diethyl ether; (D) 0.75 equiv diethyl ether; (E) 1.0 equiv diethyl ether.

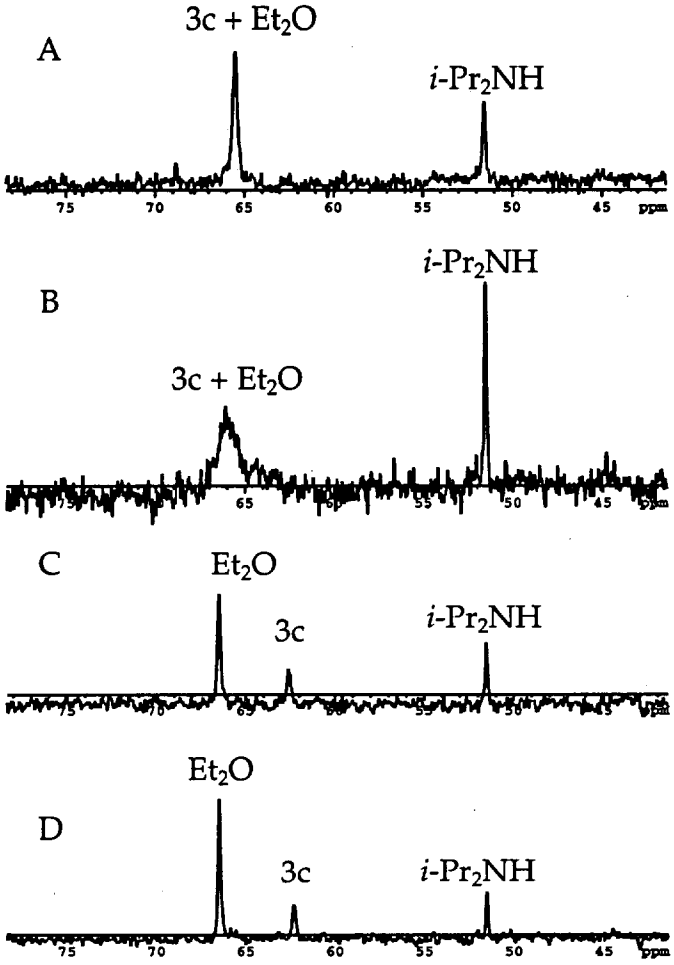

Figure XIX. ^{13}C NMR spectra of 0.1 M [^{6}Li,^{15}N]*i*-Pr_2NLi in 3:2 pentane:toluene with 2 equiv of diethyl ether at: (A) -75 °C; (B) -90 °C; (C) -115 °C; (D) -127 °C.

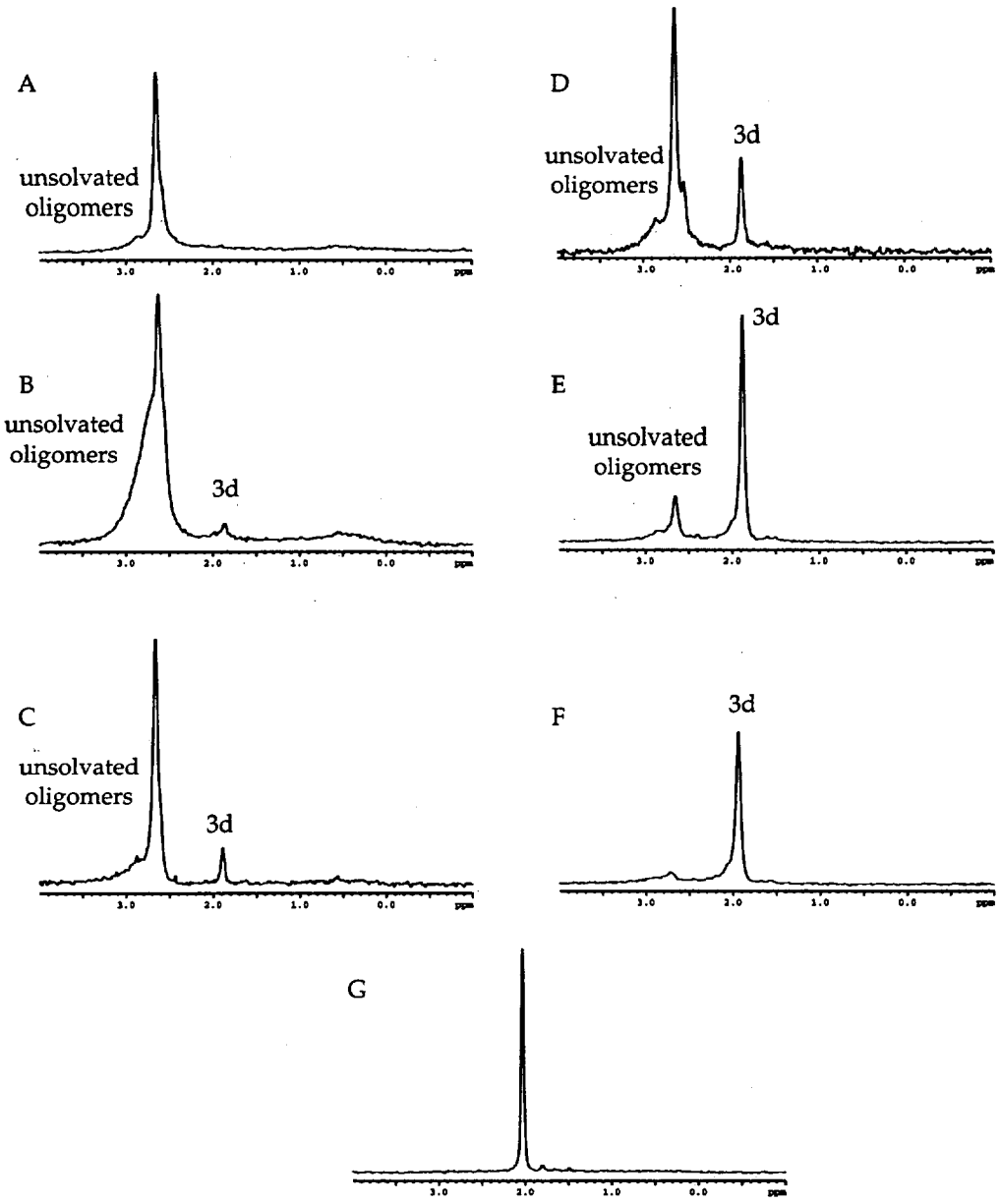

Figure XX. ^{6}Li NMR spectra of 0.1 M *i*-Pr_2NLi in 3:2 pentane:toluene: (A) $^{6}Li\{^{15}N\}$ spectrum of [^{6}Li,^{15}N]*i*-Pr_2NLi with no added ligand; (B) $^{6}Li\{^{15}N\}$ spectrum of [^{6}Li,^{15}N]*i*-Pr_2NLi with 0.5 equiv diisopropylamine; (C) $^{6}Li\{^{15}N\}$ spectrum of [^{6}Li,^{15}N]*i*-Pr_2NLi with 1.0 equiv diisopropylamine; (D) ^{6}Li spectrum of [^{6}Li]*i*-Pr_2NLi with 2.0 equiv diisopropylamine; (E) ^{6}Li spectrum of [^{6}Li]*i*-Pr_2NLi with 10 equiv diisopropylamine; (F) ^{6}Li spectrum of [^{6}Li] *i*-Pr_2NLi with 20 equiv diisopropylamine; (G) $^{6}Li\{^{15}N\}$ spectrum of [^{6}Li,^{15}N]*i*-Pr_2NLi with 1 equiv diisopropylamine and 1 equiv THF.