NMR Spectroscopic Studies of Lithium Diethylamide: Insights into Ring Laddering

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Supporting Information

XV. ^{6}Li and ^{15}N NMR spectra of 0.15 M [^{6}Li,^{15}N]Et_2NLi in 3:2 pentane:toluene with 0.75 equiv of oxetane at -125 °C: ^{15}N decoupling.

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XXVIII. ^{6}Li,^{15}N-heteronuclear multiple quantum correlation (HMQC) spectrum of 0.1 M [^{6}Li,^{15}N]Et_2NLi in 3:2 pentane:toluene with 1 equiv of THF at -87 °C.

XXIX. ^{6}Li and ^{15}N NMR spectra of 0.1 M [^{6}Li,^{15}N]Et_2NLi in diethyl ether/toluene mixtures.

XXX. ^{6}Li-detected ^{15}N zero-quantum NMR spectrum of 0.1 M [^{6}Li,^{15}N]Et_2NLi in neat diethyl ether.

I. Preparation of [^{6}Li,^{15}N]Et_2NLi

[^{15}N]Benzamide.[1] A 100 mL pear-shaped flask chilled in a 0 °C bath was charged sequentially with [^{15}N]NH_4Cl (3.1g, 57 mmol), water (10 mL), diethyl ether (15 mL), and benzoyl chloride (15.0 mL, 129 mmol). A chilled (5 °C) 19 M aqueous solution of NaOH (20 mL, 375 mmol) was added slowly via syringe directly to the aqueous layer causing precipitation of a white solid. The solid was filtered, washed repeatedly with ether and water to remove any excess benzoyl chloride and benzoic acid by-product, and dryed *in vacuo* to afford 5.15 g of [^{15}N]benzamide (76% yield). ^{1}H NMR ($CDCl_3$/DMSO) δ 7.94 (d, J_{15N-1H} = 88.4 Hz, 1H), 7.91 (d, J = 7.5 Hz, 2H), 7.49 (t, J = 6.7 Hz, 1H), 7.42 (t, J= 6.7 Hz, 2H), 7.20 (d, J_{15N-1H} = 87.7 Hz, 1H). ^{13}C{^{1}H}NMR ($CDCl_3$/DMSO) δ 166.8 (d, $J_{15N-13C}$ = 16.2 Hz), 132.5 (d, $J_{15N-13C}$ = 7.7 Hz), 129.4, 126.3, 125.8.

[^{15}N]Diethylbenzamide.[2] A 100 mL round bottom flask was charged sequentially with ^{15}N-benzamide (3.83 g, 31.4 mmol), toluene (45 mL), K_2CO_3 (6.4 g), *t*-butyl ammonium hydrogen sulfate (1.07 g), and finely ground NaOH (6 g). The heterogenous mixture was heated to 40 °C and bromoethane (6.0 mL, 80.0 mmol) in 10 mL toluene was added slowly via syringe. The mixture was refluxed with stirring for four hours. The organic phase was washed with water (2 x 100 mL), dried over Na_2SO_4, and concentrated *in vacuo*. The crude product was purified by flash chromatography (1:1 EtOAc/hexane), yielding 3.79 g (68% yield) of [^{15}N]diethylbenzamide. ^{1}H NMR ($CDCl_3$) δ 7.37 (m, 5H), 3.6-3.3 (m, 4H), 1.2-1.1 (m, 6H). ^{13}C{^{1}H}NMR ($CDCl_3$) δ 171.2 (d, $J_{15N-13C}$ = 14.8 Hz), 137.2 (d, $J_{15N-13C}$ = 7.4 Hz), 129.0, 128.3, 126.2, 43.2, 39.2, 14.1, 12.8.

[^{15}N]Diethylbenzylamine.[3] A nitrogen-flushed, 100 mL round bottom flask was charged with [^{15}N]diethylbenzamide (3.50 g, 19.7 mmol) and toluene (40 mL) and cooled to O °C. Borane-methylsulfide complex (2.0 M) in THF (6.4 mL, 12.8 mmol) was added slowly via syringe. Following a reflux for 24 hours, 25 mL of saturated $NaHCO_3$ solution was added, and the resulting solution was heated under reflux for 30 minutes. The layers were separated and the organic layer was dried over Na_2SO_4. The crude product was distilled in vacuo to give 2.45 g (76% yield) of [^{15}N]diethylbenzylamine. ^{1}H NMR ($CDCl_3$) δ 7.3 - 7.2 (m, 5H), 3.56 (m, 2H), 2.52 (q, J = 7.0 Hz, 4H), 1.04 (td, $J_{^1H-^1H}$ = 7.2 Hz, $J_{^{15}N-^1H}$ = 2.7 Hz, 6H). $^{13}C\{^1H\}$NMR ($CDCl_3$) δ 140.1 (d, $J_{^{15}N-^{13}C}$ = 1.8 Hz), 129.1, 128.3, 126.8, 57.7 (d, $J_{^{15}N-^{13}C}$ = 4.2 Hz), 46.9 (d, $J_{^{15}N-^{13}C}$ = 4.1 Hz), 11.9 (d, $J_{^{15}N-^{13}C}$ = 2.0 Hz).

[^{15}N]Diethylamine Hydrobromide.[4] A Parr hydrogenator flask was charged with [^{15}N]diethylbenzylamine (4.0 g, 24.4 mmol), acetic acid (20 mL), methanol (20 mL), and $Pd(OH)_2$ (0.380 g). The reaction vessel was placed under 50 psi H_2 for 3 days. The resulting reaction mixture was filtered through Celite, and the filtrate was heated under reflux with 5 mL of 48% HBr [5] for two hours. The solution was placed under vacuum, and the liquid was removed to give the amine hydrobromide as a pale peach solid. The solid was recrystallized in THF and a minimal amount of isopropanol to give 2.09 g (56% yield) of [^{15}N]diethylamine·HBr. ^{1}H ($CDCl_3$) δ 9.05 (d, $J_{^{15}N-^1H}$ = 73.1 Hz, 2H), 3.09 (m, 4H), 1.53 (td, $J_{^1H-^1H}$ = 7.5 Hz, $J_{^{15}N-^1H}$ = 3.5 Hz, 6H). $^{13}C\{^1H\}$NMR ($CDCl_3$) δ 42.5, 11.4.

[^{6}Li,^{15}N]Lithium Diethylamide. A 100 mL round bottom flask containing [^{15}N]diethylamine·HBr (2.09 g, 13.6 mmol) and a grain of phenanthroline was flushed with argon and charged with Et_2O (15 mL) and TDA-1[6] (5.6 mL, 17.7 mmol). (The TDA-1 serves to scavenge the resulting LiBr to prevent formation of [^{15}N]Et_2NH·LiBr.) *n*-BuLi in hexanes (1.6 M) was added with stirring to the mixture until the solution remained red for longer than 15 minutes, indicating the complete liberation of free diethylamine. The solution was vacuum transferred to a collection flask. A fixed amount of octane was added as an internal standard to estimate the quantity of diethylamine by GC.[7] The yield of [^{15}N]diethylamine was estimated to be near quantitative. The [^{15}N]Et_2NH/Et_2O solution was cooled in a 0 °C bath and [^{6}Li]*n*-BuLi (5 mL, 10.5 mmol) was added to ensure an excess of diethylamine. After 3 hours of stirring at room temperature, the solvent was removed *in vacuo*. To the resulting white residue, hexanes (40 mL) and Et_2O (13 mL) were added until all of the residue dissolved. The solution was alternately evaporated and cooled until a quantity of white solid precipitated. The mixture was filtered and yielded 0.720 g (67% yield) of solvent-free [^{6}Li,^{15}N]lithium diethylamide.

Supporting References and Footnotes

1. Aubrecht, K. B.; Lucht, B. L.; Collum, D. B. *Organometallics* **1999**, *18*, 2981.

2. Gajda, T.; Zwierzak, A. *Synthesis* **1981**, 1005.

3. Bonnat, M.; Hercouet, A.; Le Corre, M. *Synth.Commun.* **1991**, *21*, 1579.

4. Hartung, W. H.; Simonoff, R. *Org.React.* **1953**, *7*, 286.

5. The hydrochloride salt was also synthesized with similar results, but $Et_2NH{\cdot}HCl$ is very hygroscopic, unlike the hydrobromide alternative. Also the LiBr salt and resulting complexes with TDA-1 are soluble in Et_2O, whereas the LiCl salt and complexes are not.

6. TDA-1 (*tris*[2-(2-methoxyethoxy)ethyl] amine, Aldrich).

7. In subsequent studies liberating other simple dialkylamines, it was found that recrystallized *n*-BuLi in pentane solution (rather than commercial *n*-BuLi in hexanes) worked best to quantify the amount of amine liberated.

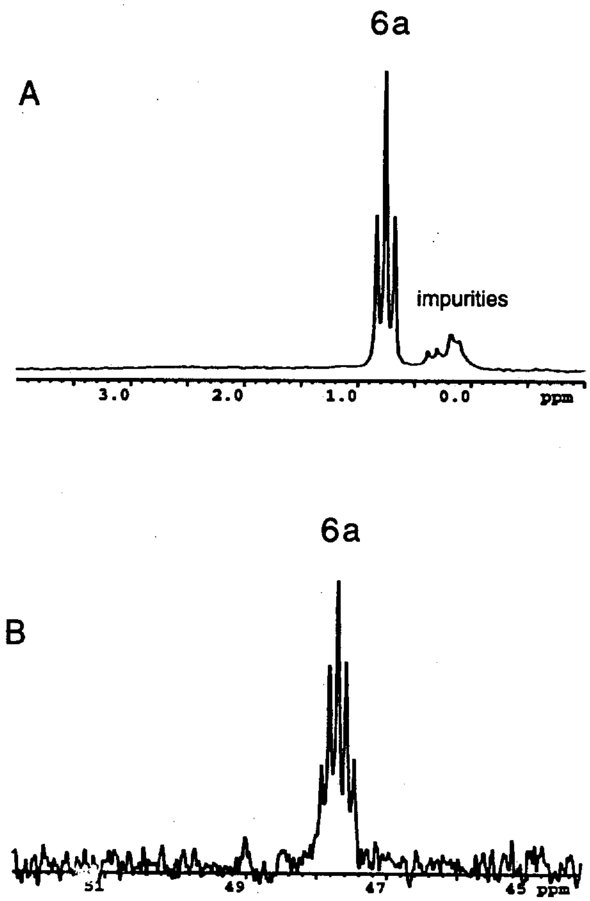

Figure II. ^{6}Li and ^{15}N NMR spectra of 0.1 M [^{6}Li,^{15}N]Et_2NLi in neat oxetane at -110 °C: (A) ^{6}Li NMR spectrum; (B) $^{15}N\{^1H\}$ NMR spectrum.

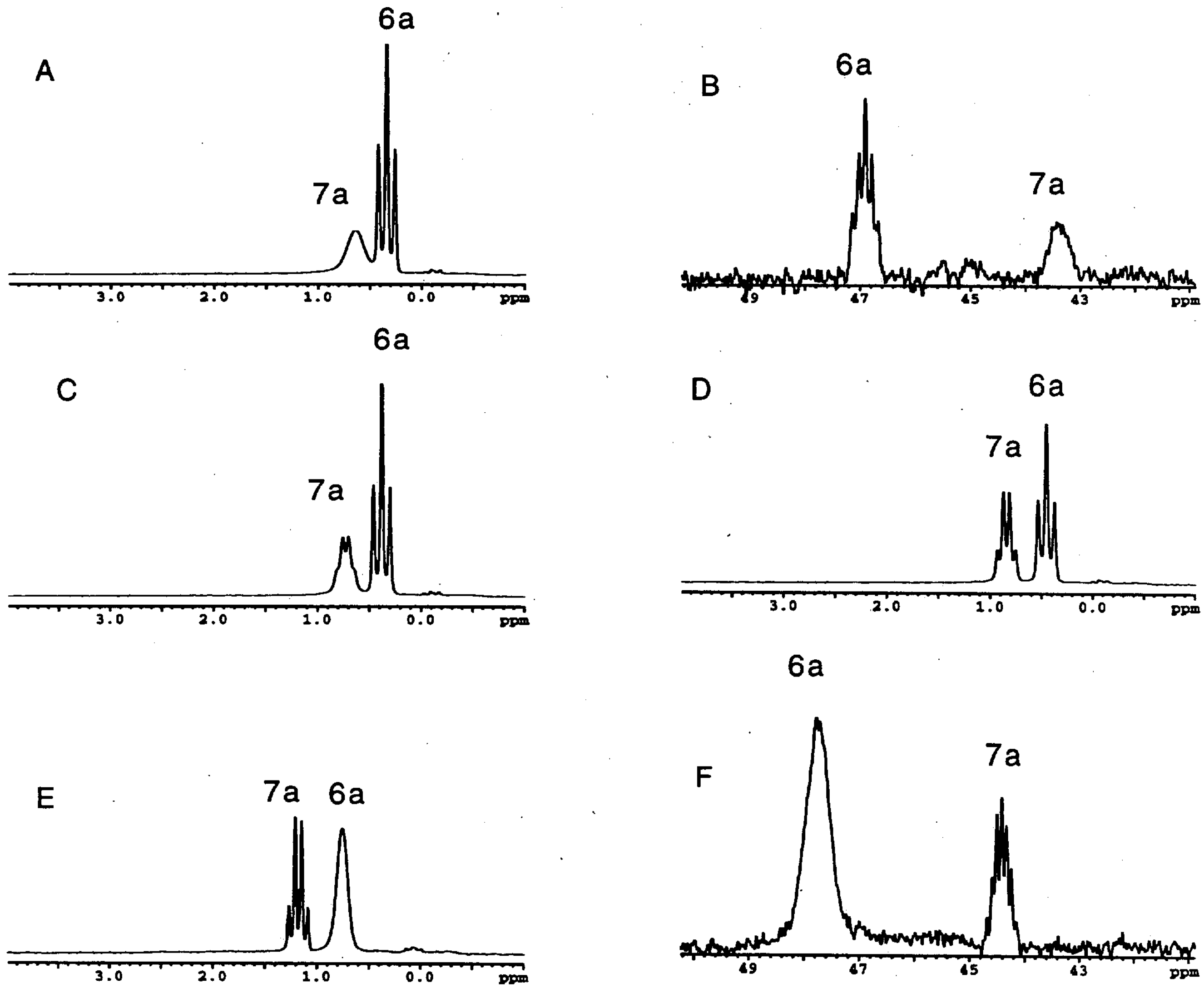

Figure III. ^{6}Li and ^{15}N NMR spectra of 0.1 M [^{6}Li,^{15}N]Et_2NLi in 3:2 pentane:toluene with 20 equiv of oxetane: (A) ^{6}Li NMR spectrum at -120 °C; (B) ^{15}N{^{1}H} NMR spectrum at -120 °C; (C) ^{6}Li NMR spectrum at -115 °C; (D) ^{6}Li NMR spectrum at -110 °C; (E) ^{6}Li NMR spectrum at -90 °C; (F) ^{15}N{^{1}H} NMR spectrum at -90 °C.

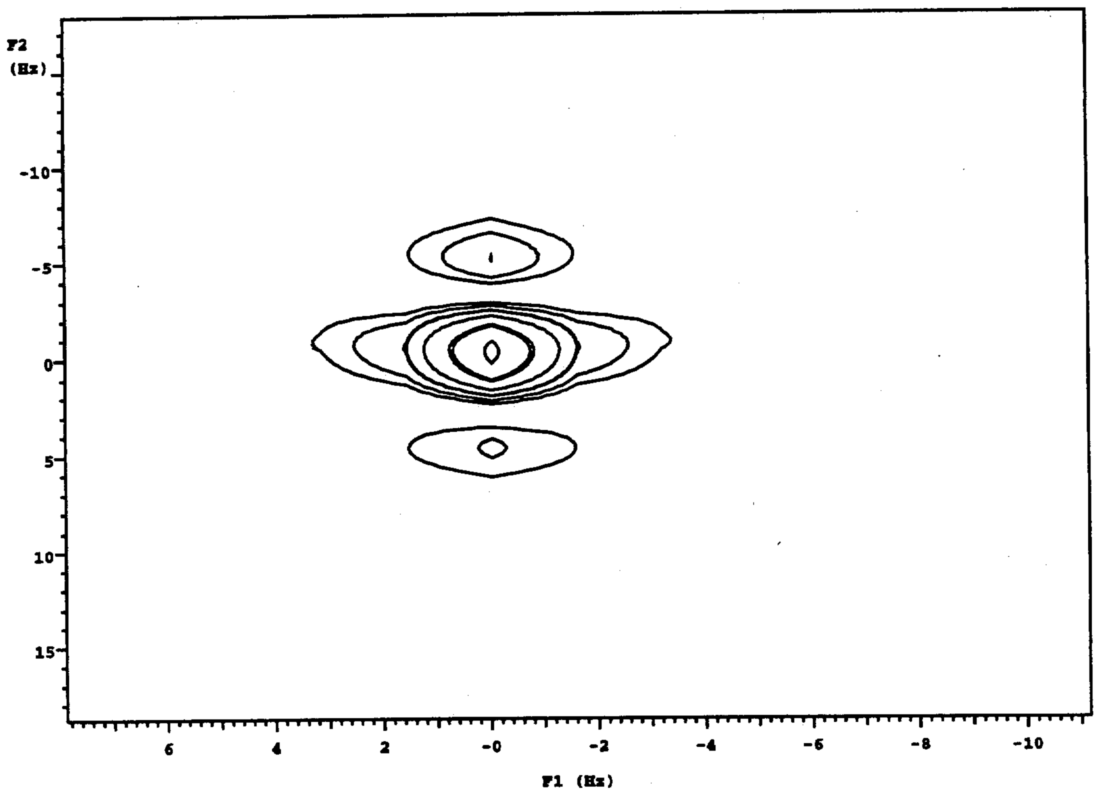

Figure IV. ^{6}Li-detected ^{15}N zero-quantum NMR spectrum of 0.1 M [^{6}Li,^{15}N]Et_2NLi in 3:2 pentane:toluene with 20 equiv of oxetane at -120 °C.

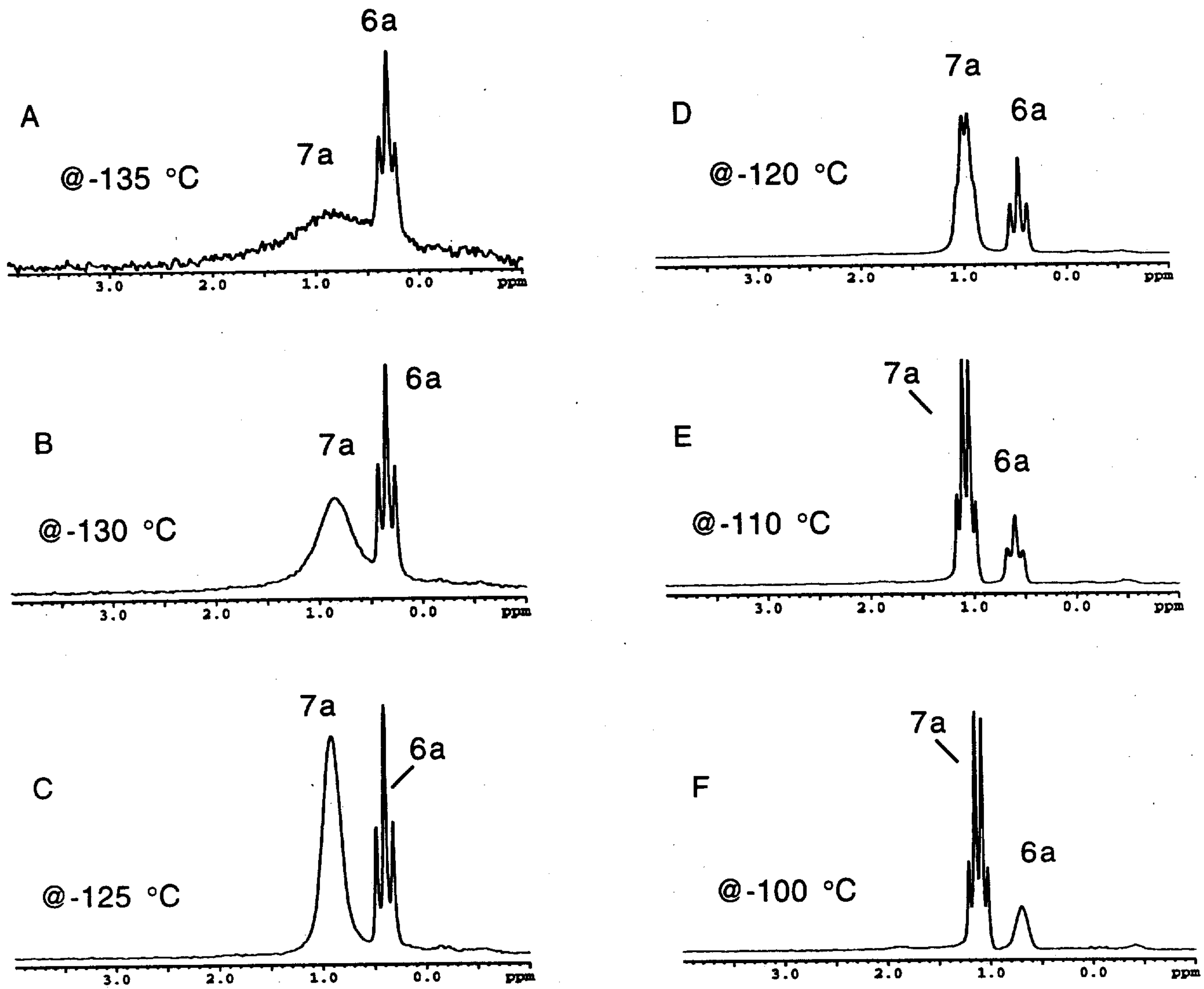

Figure V. ^{6}Li NMR spectra of 0.1 M [^{6}Li,^{15}N]Et_2NLi in 3:2 pentane:toluene with 6 equiv of oxetane at the temperatures indicated.

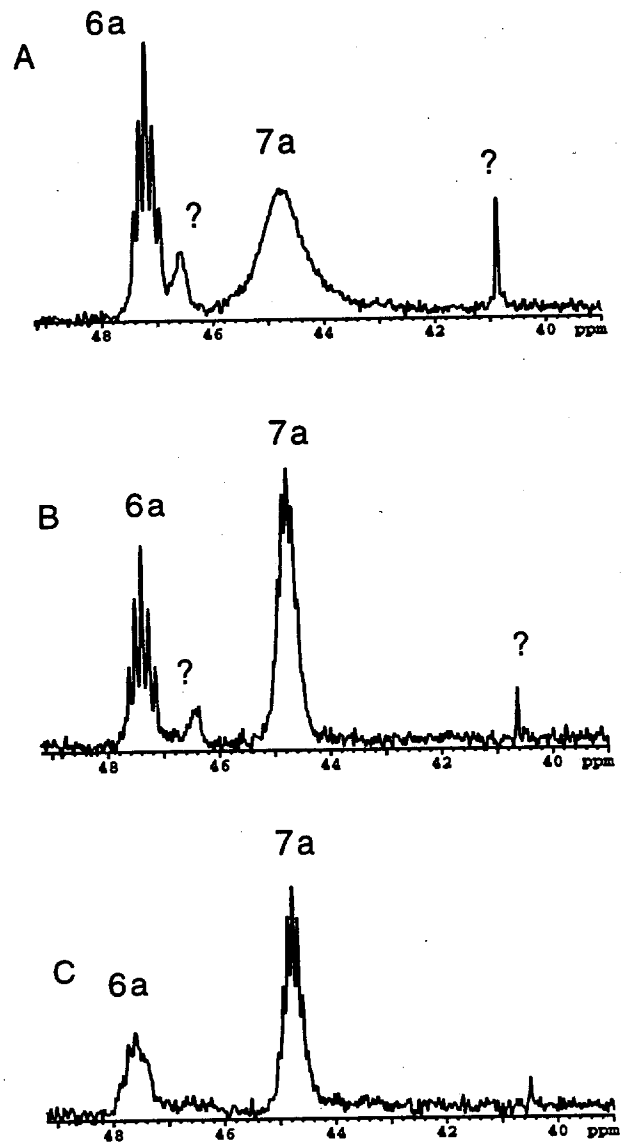

Figure VI. $^{15}N\{^{1}H\}$ NMR spectra of 0.1 M [^{6}Li,^{15}N]Et_2NLi in 3:2 pentane:toluene with 6 equiv of oxetane at : (A) -135 °C; (B) -120 °C; (C) -110 °C.

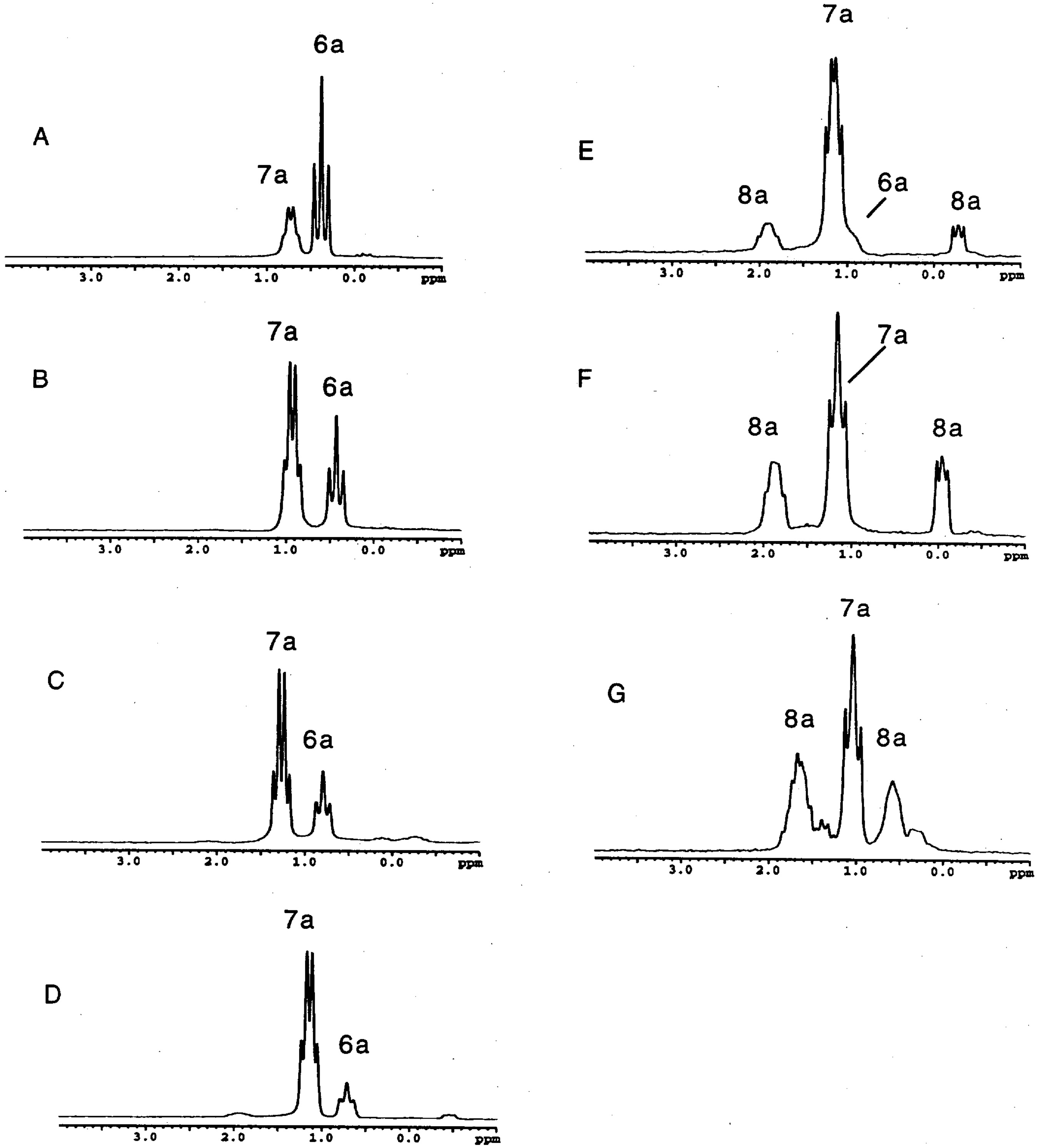

Figure VII. ^{6}Li NMR spectra of 0.1 M [^{6}Li,^{15}N]Et_2NLi in 3:2 pentane:toluene at -115 °C with: (A) 20 equiv oxetane; (B) 9 equiv oxetane; (C) 6 equiv oxetane; (D) 3 equiv oxetane; (E) 1.25 equiv oxetane; (F) 1 equiv oxetane; (G) 0.75 equiv oxetane.

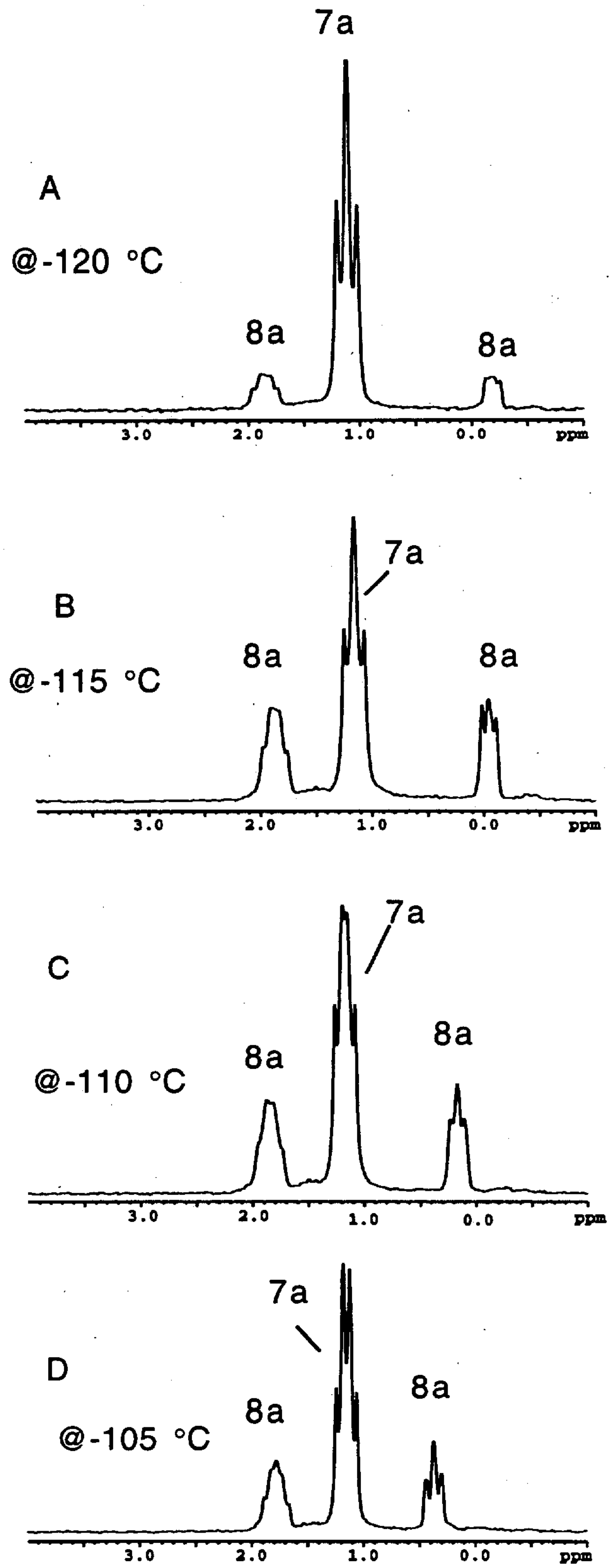

Figure VIII. ^{6}Li NMR spectra of 0.1 M [^{6}Li,^{15}N]Et_2NLi in 3:2 pentane:toluene with 1 equiv oxetane at: (A) -120°C; (B) -115°C; (C) -110°C; (D) -105°C.

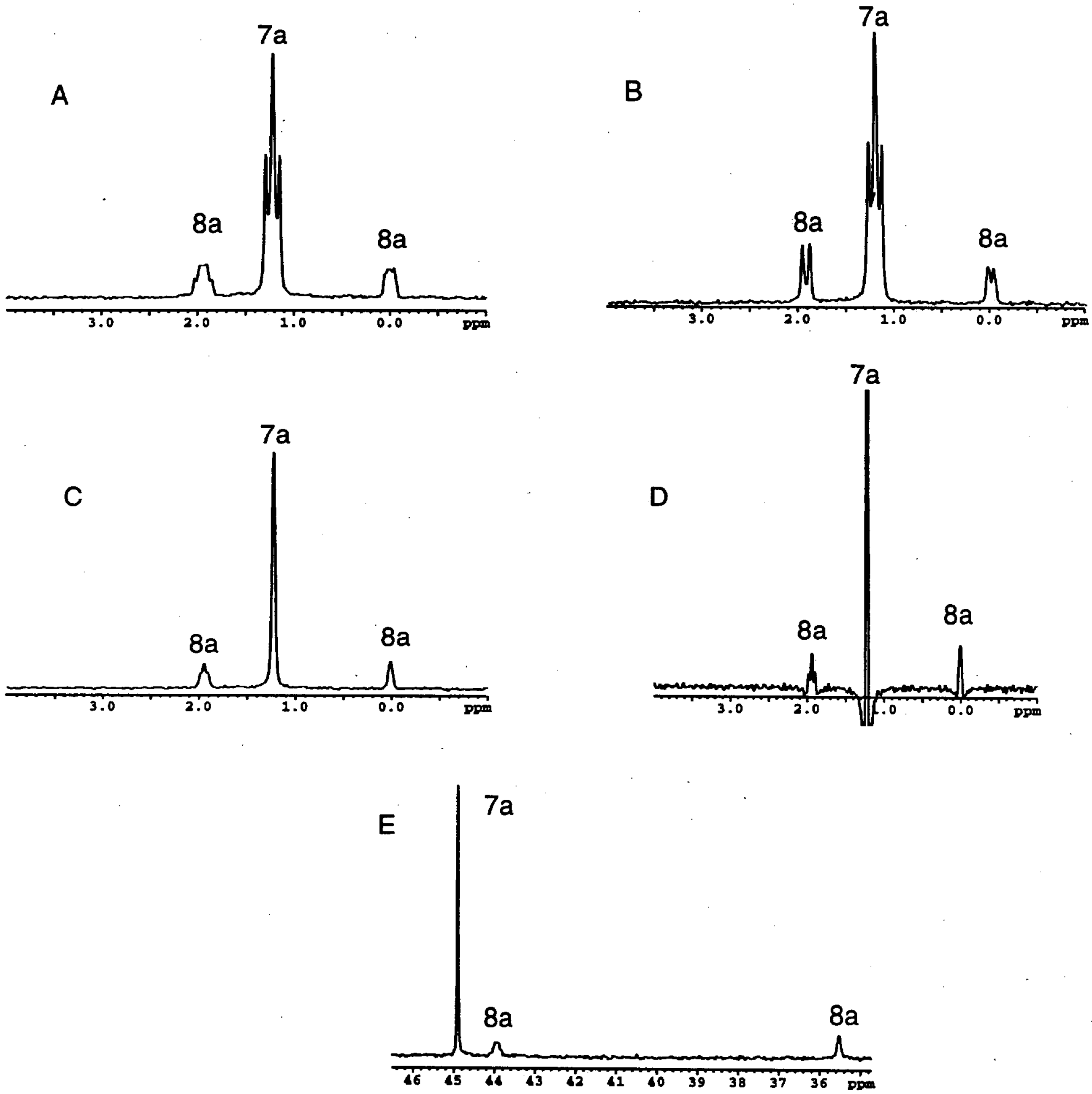

Figure IX. ^{6}Li and ^{15}N NMR spectra of 0.1 M [^{6}Li,^{15}N]Et_2NLi in 3:2 pentane:toluene with 1.0 equiv of oxetane at -125 °C: (A) ^{6}Li spectrum; (B) ^{6}Li spectrum, ^{15}N single frequency decoupled at 35.5 ppm; (C) ^{6}Li spectrum, ^{15}N single frequency decoupled at 43.9 ppm; (D) spectrum C with sine bell resolution enhancement function applied (Note: Coupling for **8a** doublet at 0.0 ppm is small and appears as a broad singlet in this spectrum. For a better view see Figure XD); (E) ^{15}N{^{1}H, ^{6}Li}spectrum.

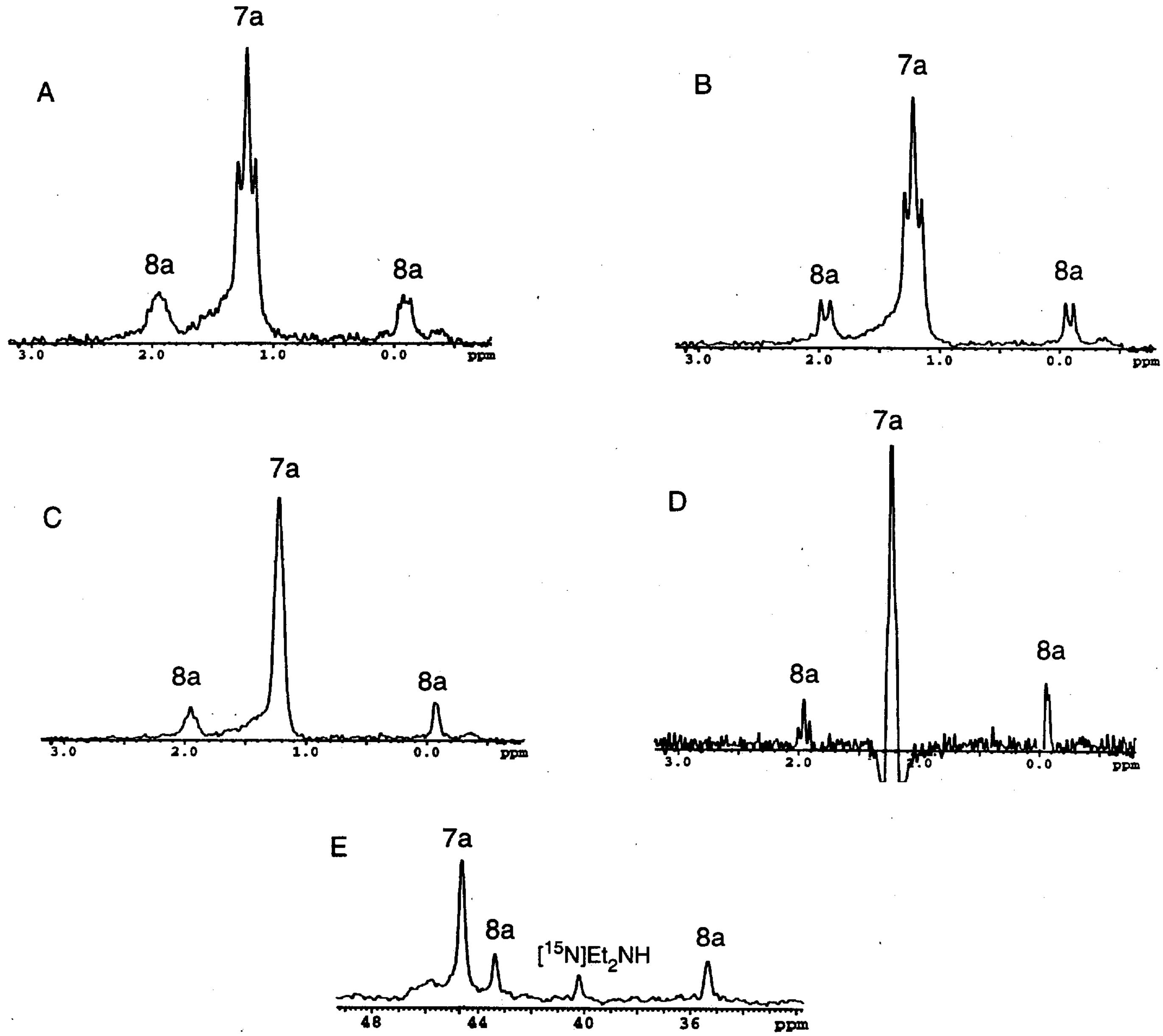

Figure X. ^{6}Li and ^{15}N NMR spectra of 0.1 M [^{6}Li,^{15}N]Et_2NLi in 3:2 pentane:toluene with 0.88 equiv of oxetane at -125 °C: (A) ^{6}Li spectrum; (B) ^{6}Li spectrum, ^{15}N single frequency decoupled at 35.3 ppm; (C) ^{6}Li spectrum, ^{15}N single frequency decoupled at 43.3 ppm; (D) spectrum C with sine bell resolution; (E) ^{15}N{^{1}H, ^{6}Li}spectrum.

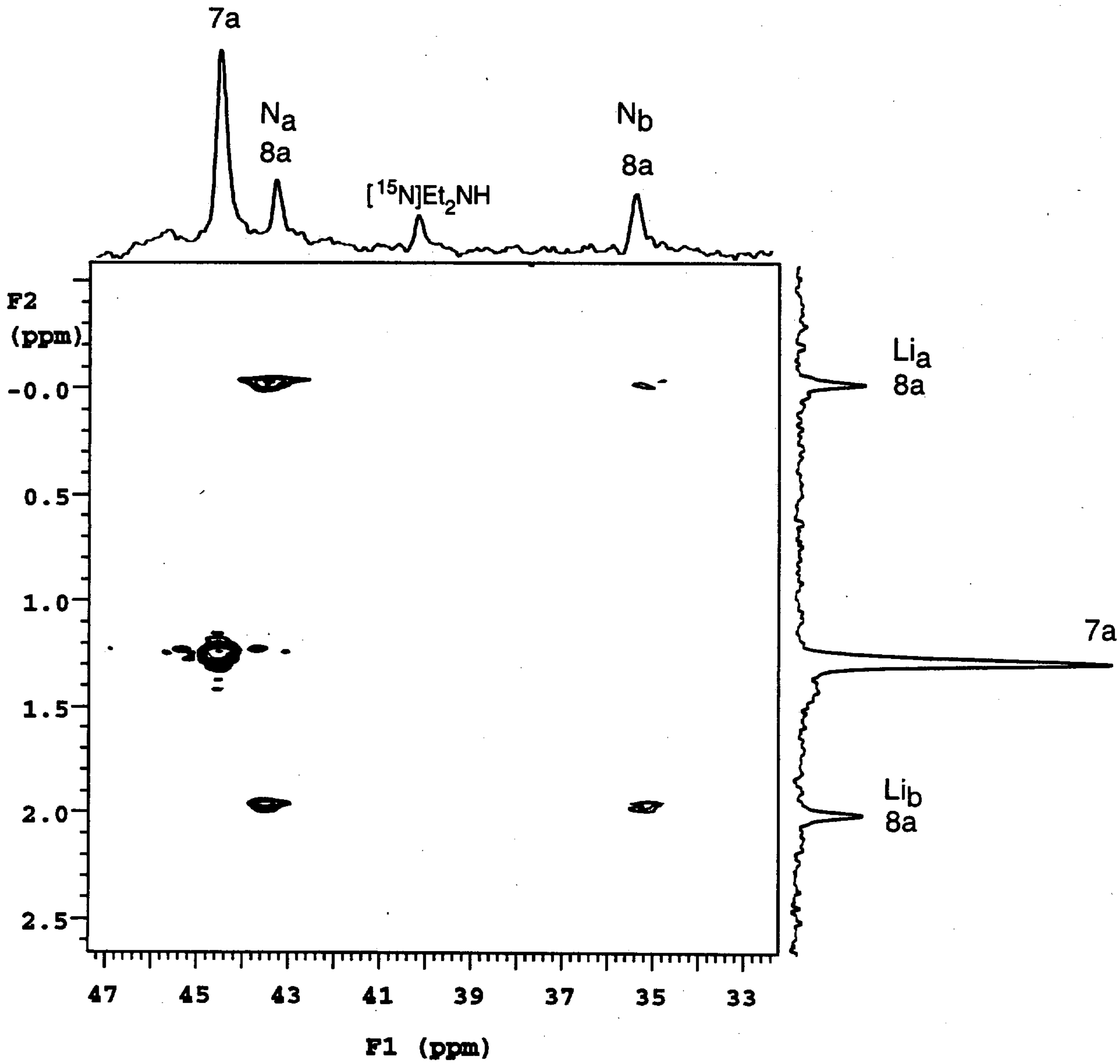

Figure XI. $^{6}Li,^{15}N$-heteronuclear multiple quantum correlation (HMQC) spectrum of 0.1 M $[^{6}Li,^{15}N]Et_2NLi$ in 3:2 pentane:toluene with 1.25 equiv of oxetane at -125 °C.

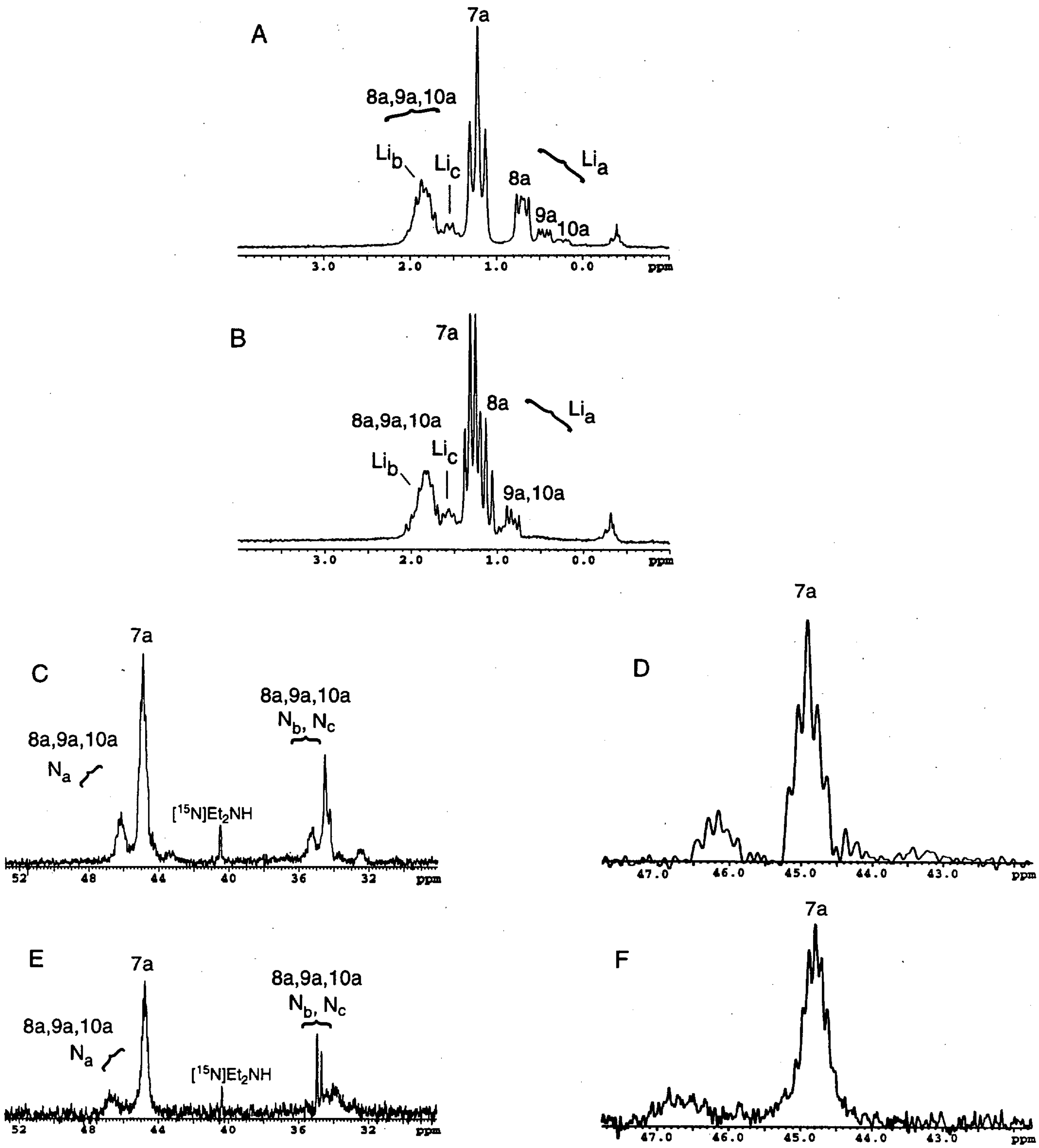

Figure XII. ^{6}Li and ^{15}N NMR spectra of 0.1 M [^{6}Li,^{15}N]Et_2NLi in 3:2 pentane:toluene with 0.75 equiv of oxetane: (A) ^{6}Li spectrum at -125 °C; (B) ^{6}Li spectrum at -105 °C; (C) $^{15}N\{^{1}H\}$ spectrum at -125 °C; (D) spectrum C expanded; (E) $^{15}N\{^{1}H\}$ spectrum at -105 °C; (F) spectrum E expanded.

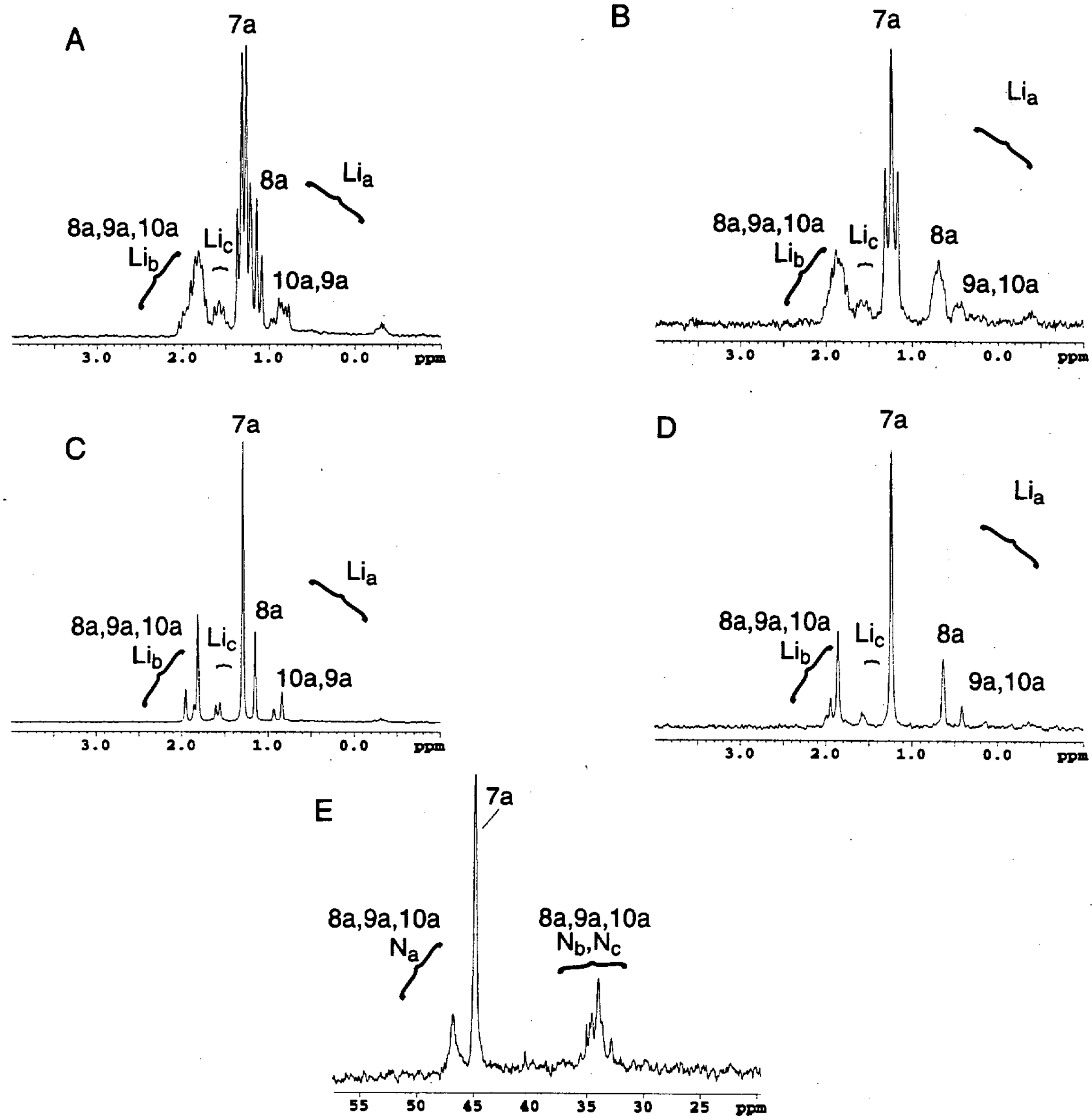

Figure XIII. ^{6}Li and ^{15}N NMR spectra of 0.15 M [^{6}Li,^{15}N]Et_2NLi in 3:2 pentane:toluene with 0.75 equiv of oxetane: (A) ^{6}Li spectrum at -105 °C; (B) ^{6}Li spectrum at -125 °C; (C) ^{6}Li{^{15}N} spectrum at -105 °C; (D) ^{6}Li{^{15}N} spectrum at -125 °C; (E) ^{15}N{^{1}H, ^{6}Li} spectrum at -105 °C.

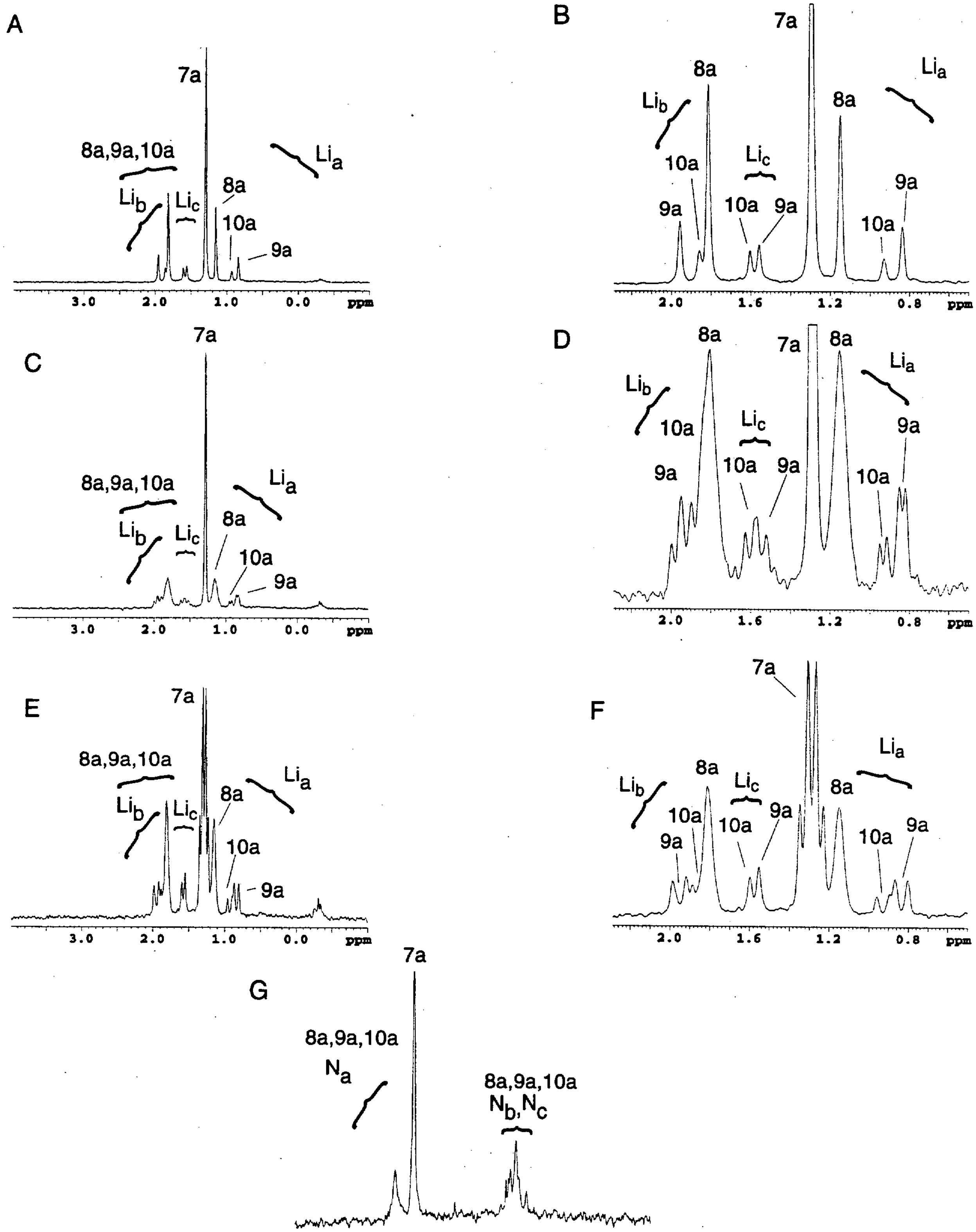

Figure XIV. ^{6}Li and ^{15}N NMR spectra of 0.15 <u>M</u> [^{6}Li,^{15}N]Et_2NLi in 3:2 pentane:toluene with 0.75 equiv of oxetane at -105 °C: (A) ^{6}Li{^{15}N} spectrum; (B) spectrum A expanded; (C) ^{6}Li spectrum, ^{15}N single frequency decoupled at 44.8 ppm; (D) spectrum B expanded; (E) ^{6}Li spectrum, ^{15}N single frequency decoupled at 32.8 ppm; (F) spectrum E expanded; (G) ^{15}N{^{1}H,^{6}Li} spectrum.

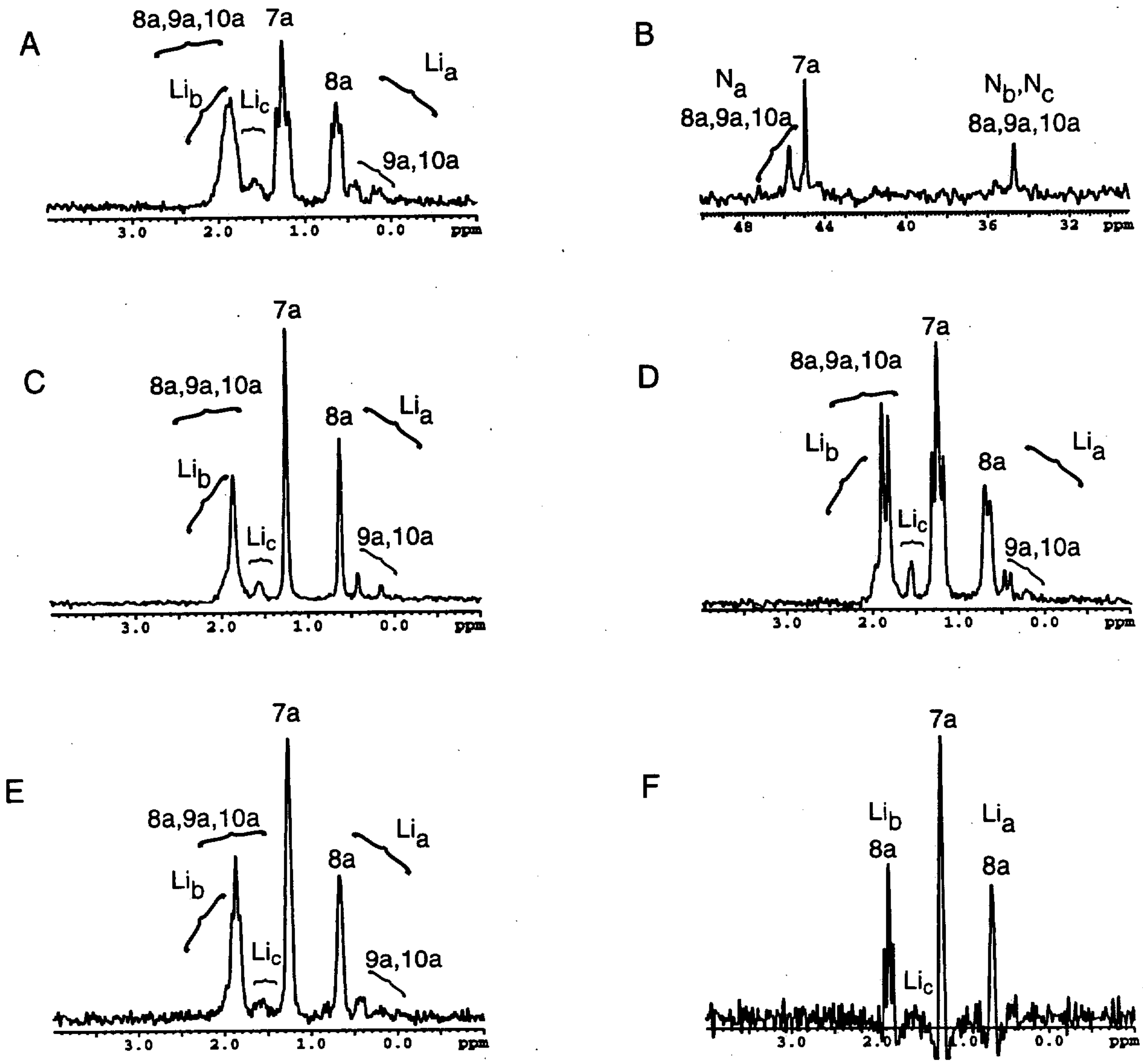

Figure XV. ^{6}Li and ^{15}N NMR spectra of 0.1 M [^{6}Li,^{15}N]Et_2NLi in 3:2 pentane:toluene with 0.75 equiv of oxetane at -125 °C: (A) ^{6}Li spectrum; (B) ^{15}N{^{1}H, ^{6}Li} spectrum; (C) ^{6}Li{^{15}N} spectrum; (D) ^{6}Li spectrum, ^{15}N single frequency decoupled at 34.7 ppm; (E) ^{6}Li spectrum, ^{15}N single frequency decoupled at 45.7 ppm; (F) spectrum E with sine bell resolution enhancement function applied.

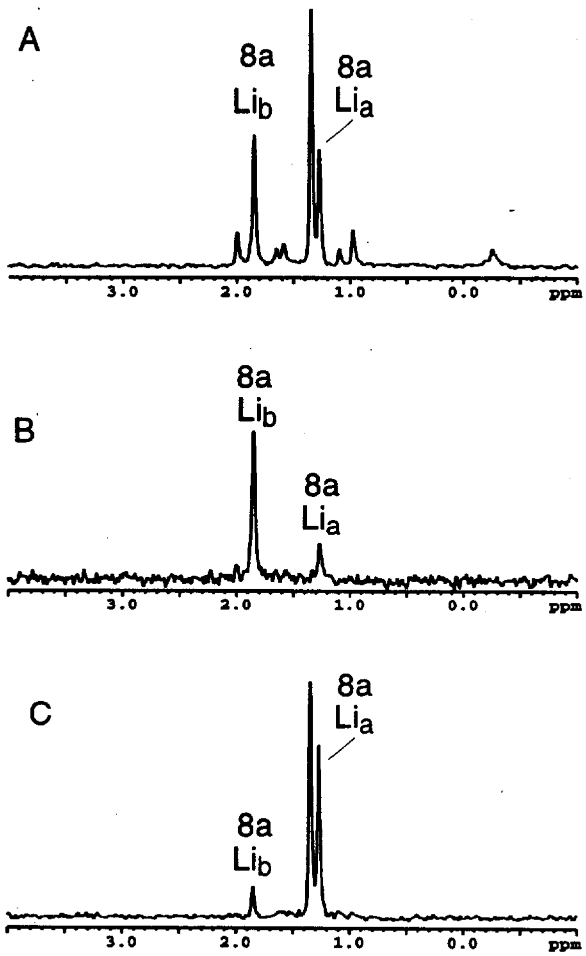

Figure XVI. ^{6}Li NMR spectra of 0.15 M [^{6}Li,^{15}N]Et$_2$NLi in 3:2 pentane:toluene with 0.75 equiv of oxetane at -100 °C: (A) ^{6}Li{^{15}N} spectrum; (B) ^{6}Li one-dimensional exchange difference spectrum, selectively irradiated at 1.84 ppm, $\tau_m = 0.4$ s; (C) ^{6}Li one-dimensional exchange difference spectrum, selectively irradiated at 1.27 ppm, $\tau_m = 0.4$ s.

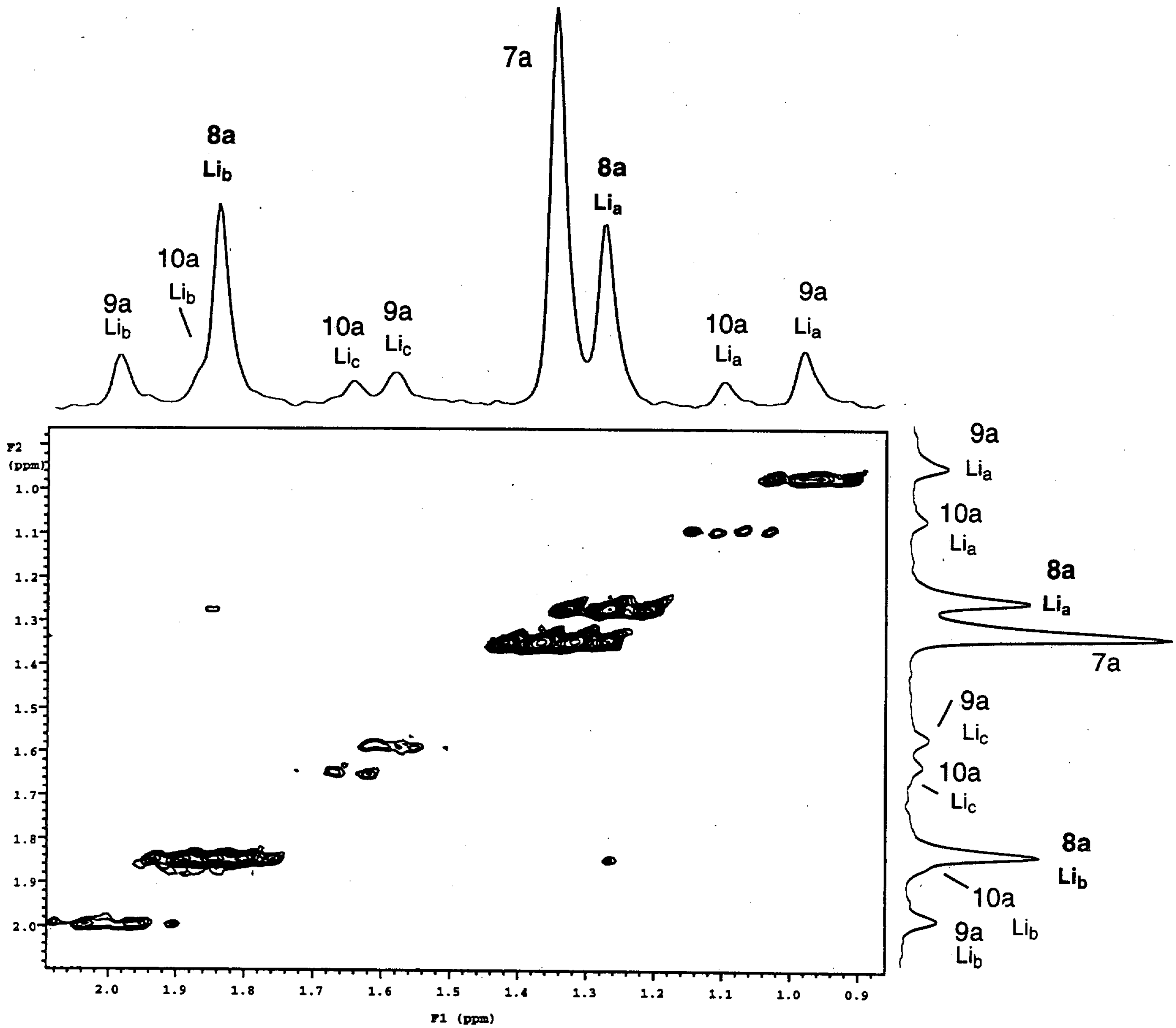

Figure XVII. ^{6}Li-^{6}Li exchange (EXSY) spectrum of 0.1 M [^{6}Li,^{15}N]Et_2NLi in 3:2 pentane:toluene with 0.75 equiv of oxetane at -100 °C, $\tau_m = 0.6$ s.

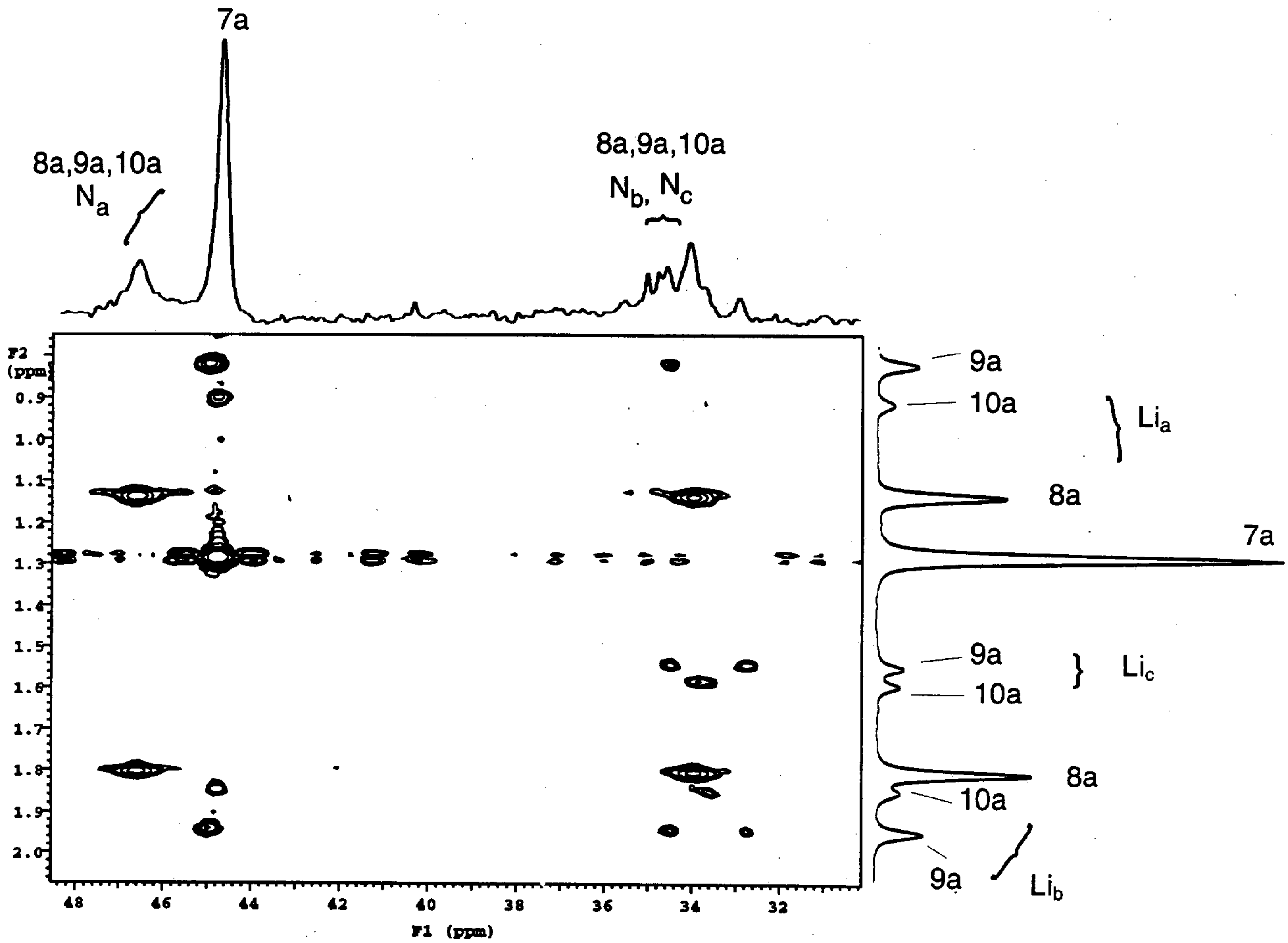

Figure XVIII. ^{6}Li,^{15}N-heteronuclear multiple quantum correlation (HMQC) spectrum of 0.15 M [^{6}Li,^{15}N]Et_2NLi in 3:2 pentane:toluene with 0.75 equiv of oxetane at -105 °C.

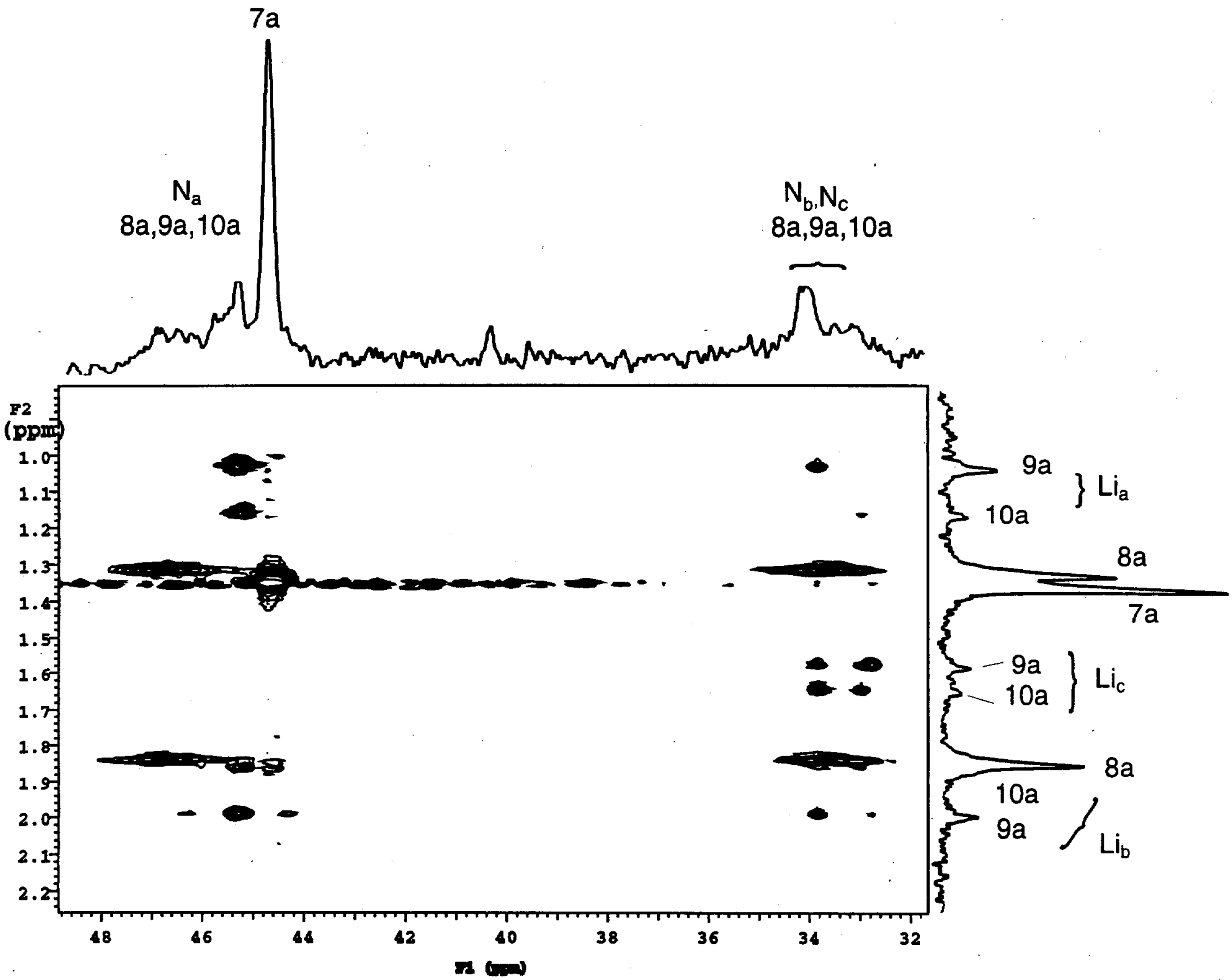

Figure XIX. ^{6}Li,^{15}N-heteronuclear multiple quantum correlation (HMQC) spectrum of 0.15 M [^{6}Li,^{15}N]Et_2NLi in 3:2 pentane:toluene with 0.75 equiv of oxetane at -95 °C.

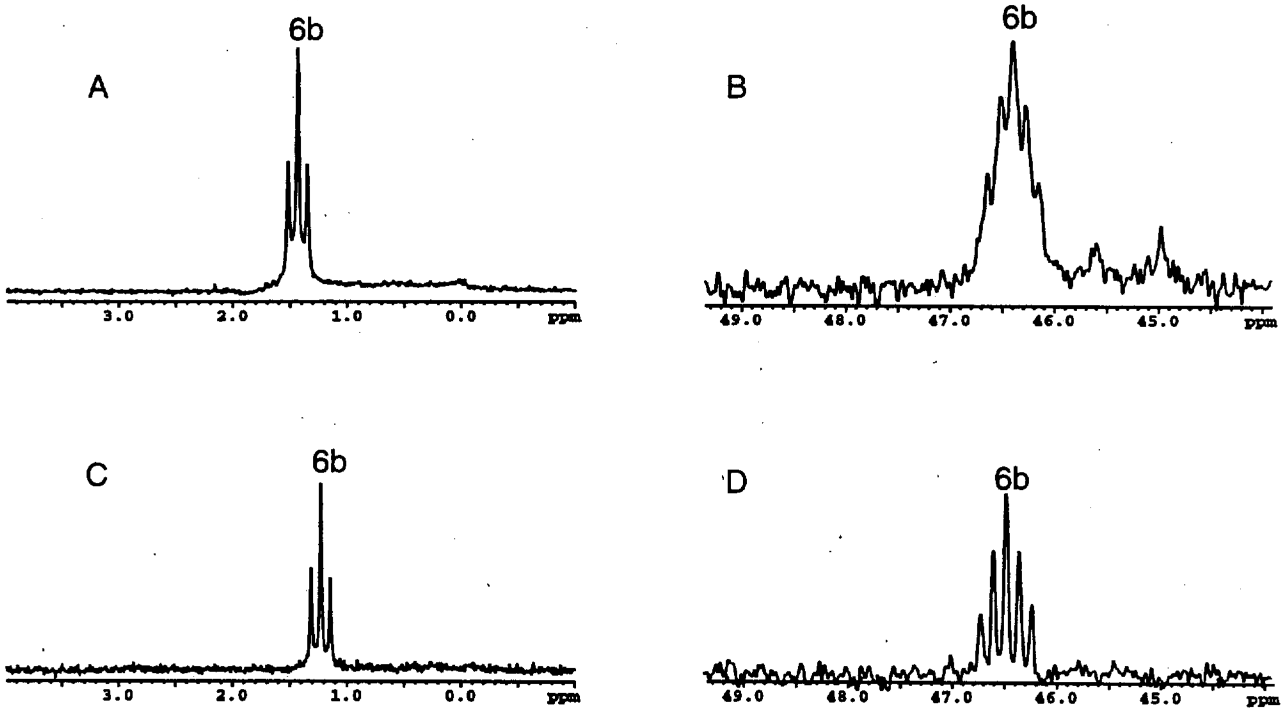

Figure XX. ^{6}Li and ^{15}N NMR spectra of 0.1 <u>M</u> [^{6}Li,^{15}N]Et_2NLi: (A) ^{6}Li spectrum in toluene with 20 equiv of THF at -110 °C; (B) $^{15}N\{^{1}H\}$ spectrum in toluene with 20 equiv of THF at -110 °C; (C) ^{6}Li spectrum in neat THF at -105 °C; (D) $^{15}N\{^{1}H\}$ spectrum in neat THF at -105 °C.

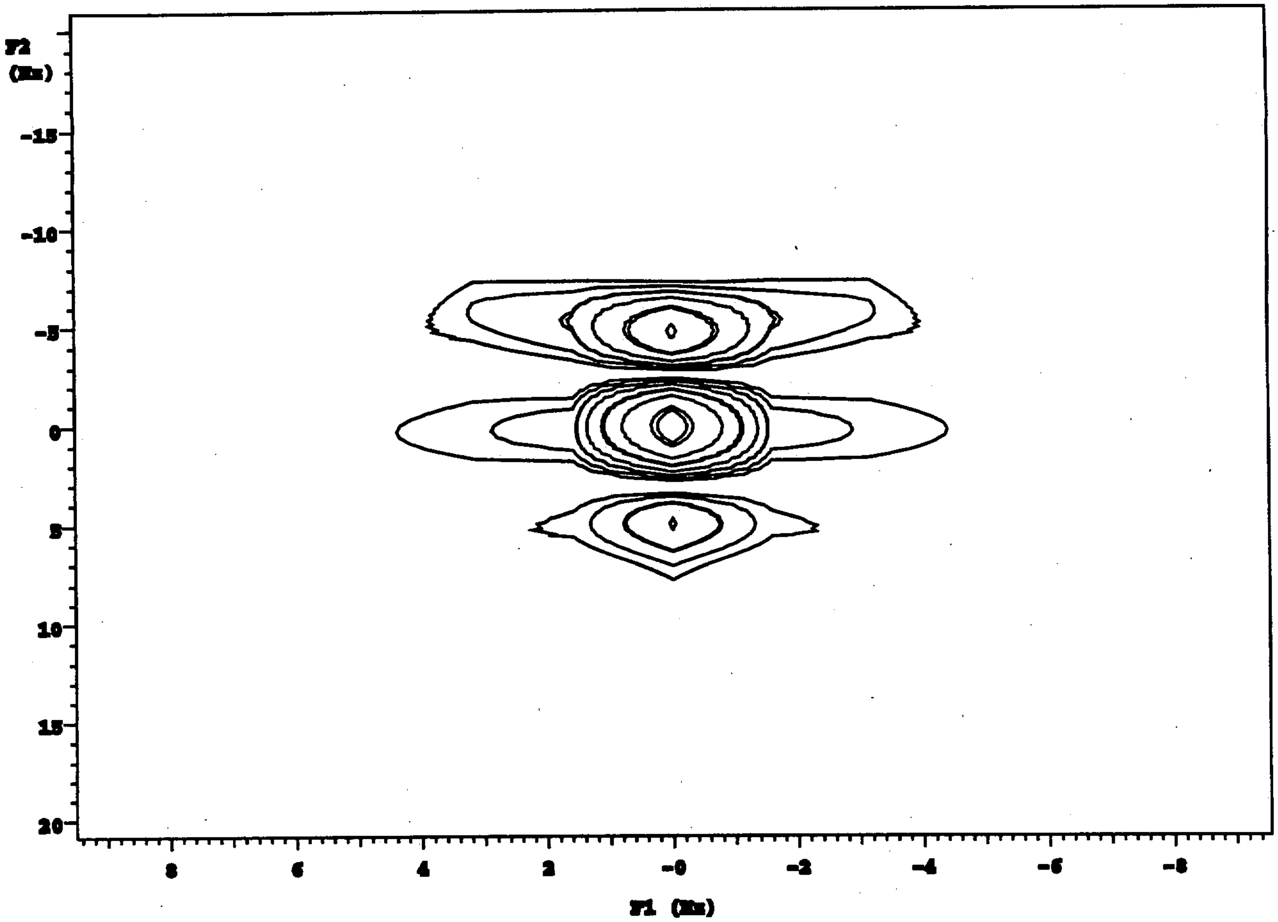

Figure XXI. ^{6}Li-detected ^{15}N zero-quantum NMR spectrum of 0.1M [^{6}Li,^{15}N]Et_2NLi in neat THF at -105 °C.

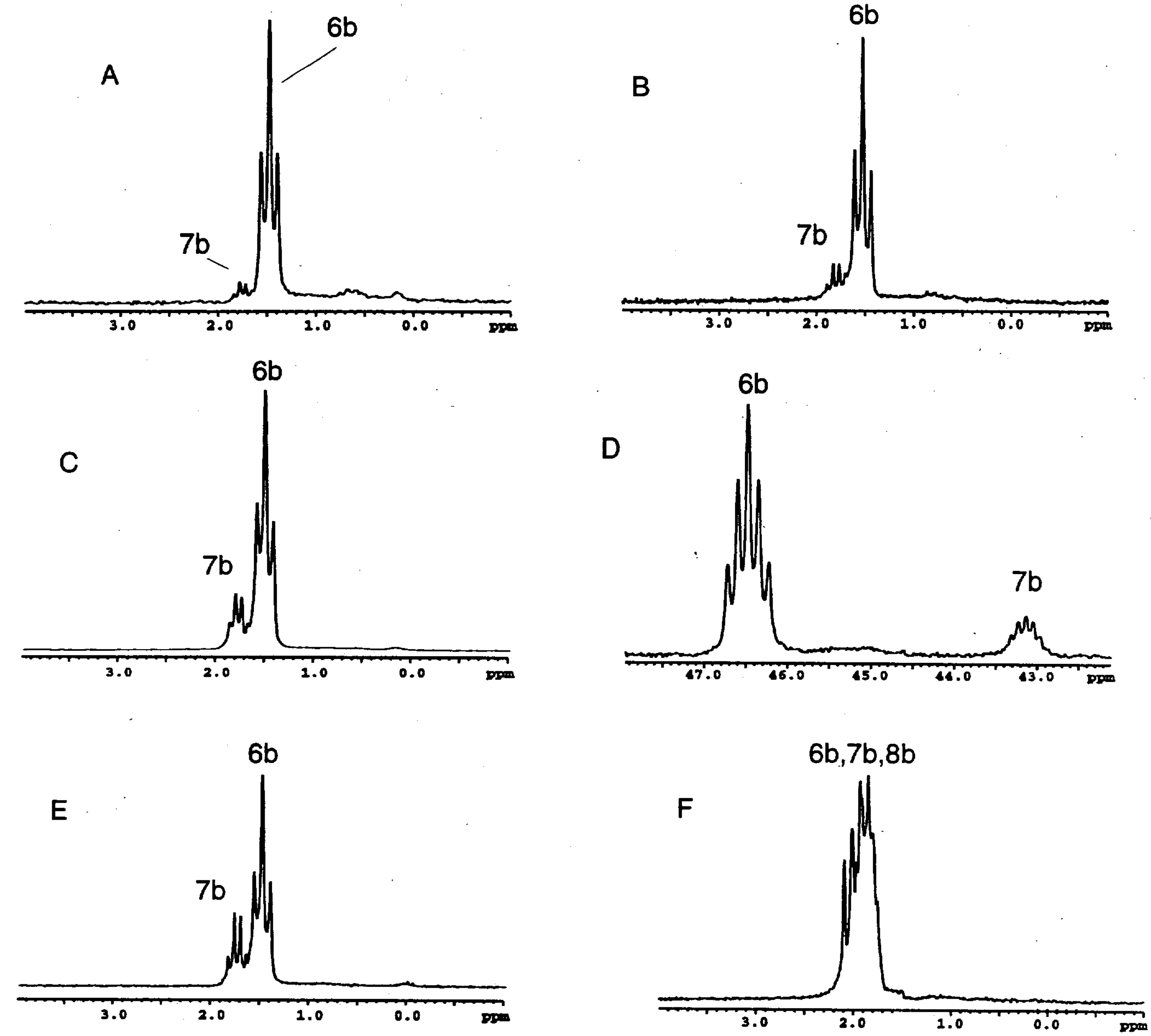

Figure XXII. ^{6}Li and ^{15}N NMR spectra of [^{6}Li,^{15}N]Et_2NLi in 3:2 pentane:toluene with THF co-solvent: (A) ^{6}Li spectrum of 0.08 M [^{6}Li,^{15}N]Et_2NLi with 9 equiv of THF at -115 °C; (B) ^{6}Li spectrum of 0.1 M [^{6}Li,^{15}N]Et_2NLi with 5 equiv of THF at -110 °C; (C) ^{6}Li spectrum of 0.2 M [^{6}Li,^{15}N]Et_2NLi with 3 equiv of THF at -115 °C; (D) ^{15}N{^{1}H} spectrum of 0.2 M [^{6}Li,^{15}N]Et_2NLi with 3 equiv of THF at -115 °C; (E) ^{6}Li spectrum of 0.16 M [^{6}Li,^{15}N]Et_2NLi with 2 equiv of THF at -115 °C; (F) ^{6}Li spectrum of 0.1 M [^{6}Li,^{15}N]Et_2NLi with 1 equiv of THF at -110 °C.

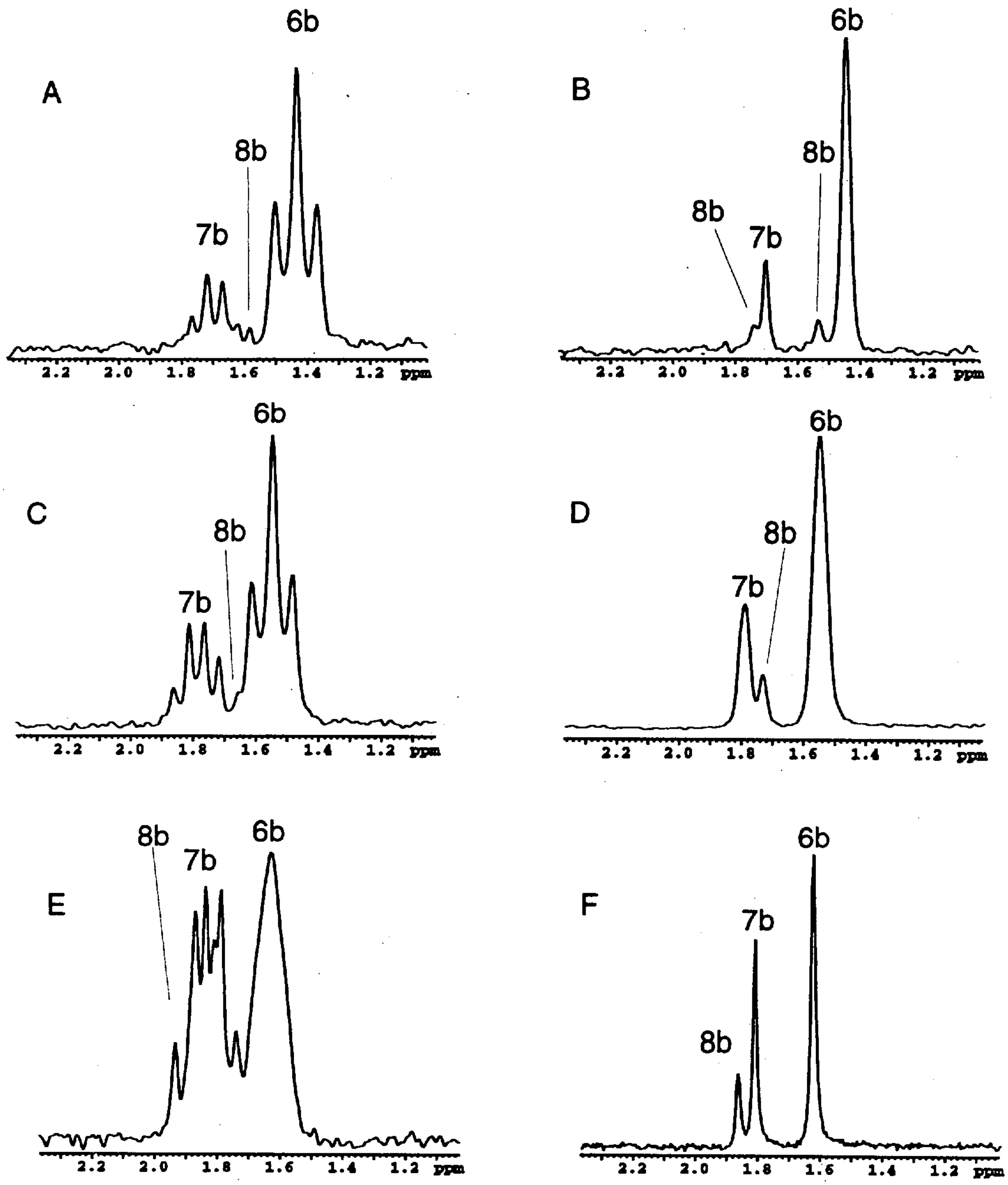

Figure XXIII. ^{6}Li NMR spectra of 0.16 M [^{6}Li,^{15}N]Et_2NLi in 3:2 pentane:toluene with 2 equiv of THF: (A) at -120 °C; (B) spectrum A broad-band decoupled; (C) at -115 °C; (D) spectrum C broad-band decoupled; (E) at -105 °C; (F) spectrum E broad-band decoupled.

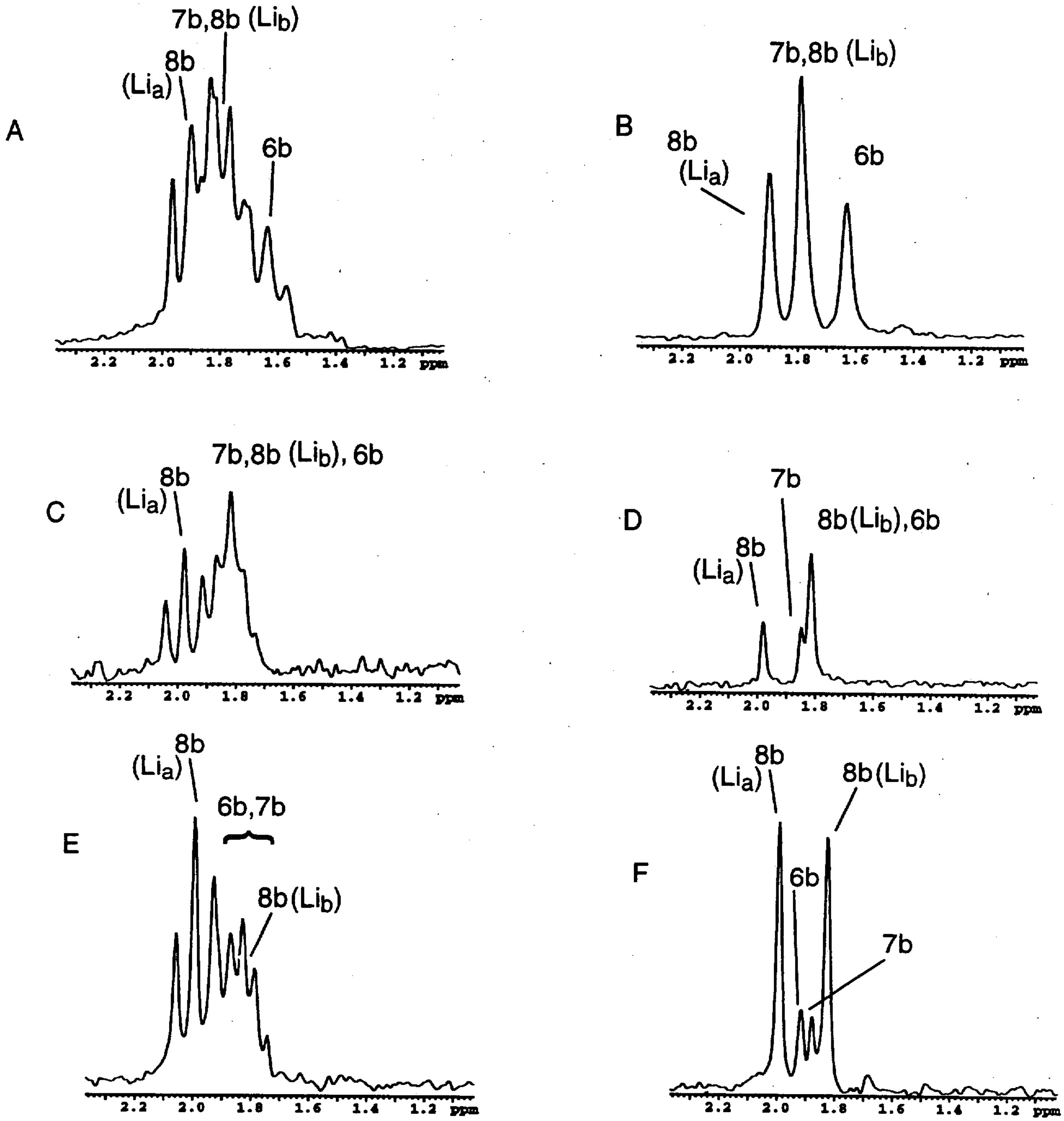

Figure XXIV. ^{6}Li NMR spectra of 0.1 M [^{6}Li,^{15}N]Et_2NLi in 3:2 pentane:toluene with 1 equiv of THF: (A) ^{6}Li spectrum at -118 °C; (B) ^{6}Li{^{15}N} spectrum at -118 °C; (C) ^{6}Li spectrum at -100 °C; (D) ^{6}Li{^{15}N} spectrum at -100 °C; (E) ^{6}Li spectrum at -87 °C; (F) ^{6}Li{^{15}N} spectrum at -87 °C.

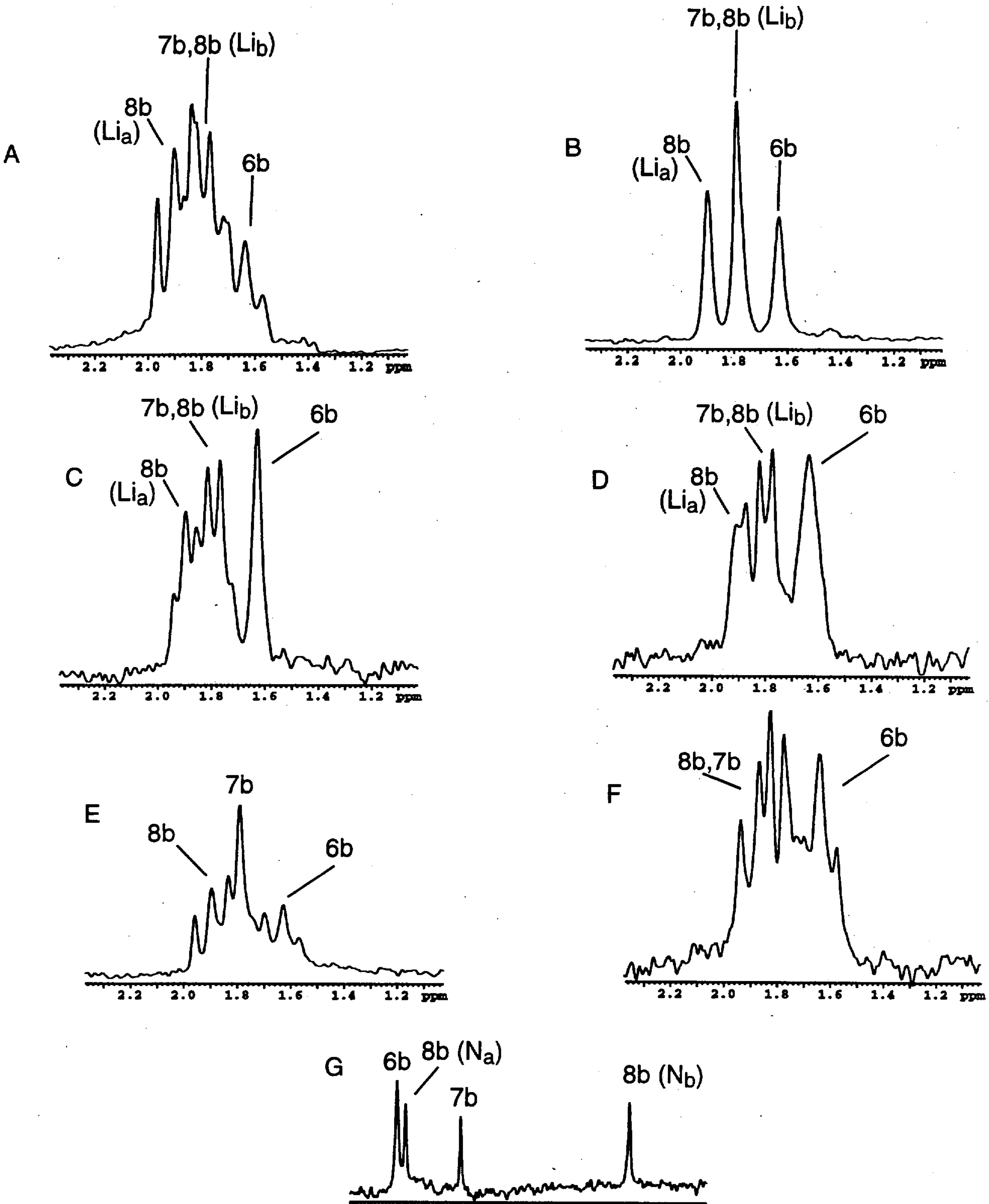

Figure XXV. ^{6}Li and ^{15}N NMR spectra of 0.1 M [^{6}Li,^{15}N]Et_2NLi in 3:2 pentane:toluene with 1.0 equiv of THF at -118 °C: (A) ^{6}Li spectrum; (B) ^{6}Li{^{15}N} spectrum; (C) ^{6}Li spectrum, ^{15}N single frequency decoupled at 46.8 ppm; (D) ^{6}Li spectrum, ^{15}N single frequency decoupled at 46.3 ppm; (E) ^{6}Li spectrum, ^{15}N single frequency decoupled at 43.2 ppm; (F) ^{6}Li spectrum, ^{15}N single frequency decoupled at 33.8 ppm; (G) ^{15}N{^{1}H, ^{6}Li} spectrum.

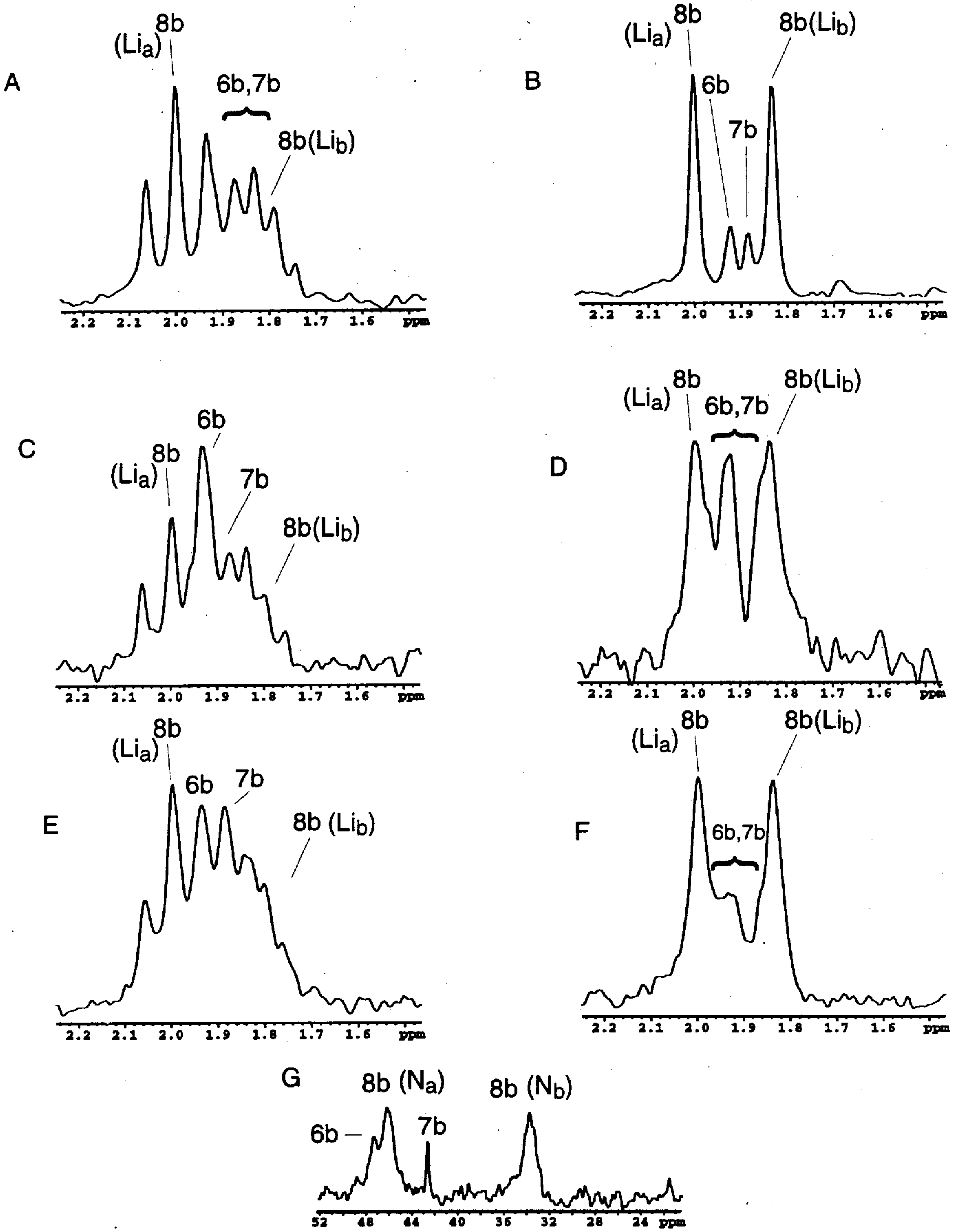

Figure XXVI. ^{6}Li and ^{15}N NMR spectra of 0.1 M [^{6}Li,^{15}N]Et$_2$NLi in 2:1 pentane:toluene with 1.0 equiv of THF at -87 °C: (A) ^{6}Li spectrum; (B) ^{6}Li{^{15}N} spectrum; (C) ^{6}Li spectrum, ^{15}N single frequency decoupled at 50.8 ppm; (D) ^{6}Li spectrum, ^{15}N single frequency decoupled at 46.0 ppm; (E) ^{6}Li spectrum, ^{15}N single frequency decoupled at 42.6 ppm; (F) ^{6}Li spectrum, ^{15}N single frequency decoupled at 33.8 ppm; (G) ^{15}N{^{1}H, ^{6}Li} spectrum.

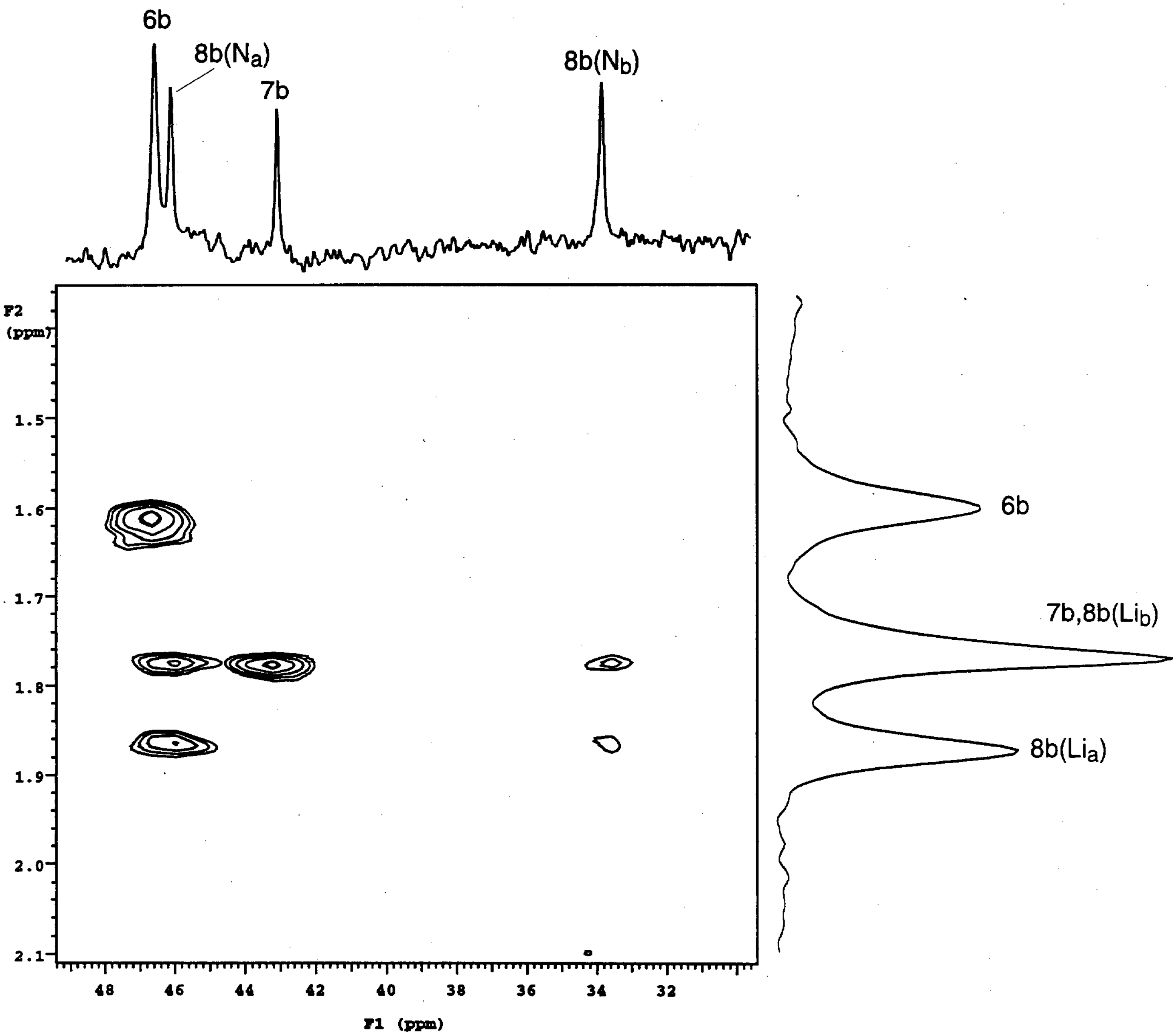

Figure XXVII. $^{6}Li,^{15}N$-heteronuclear multiple quantum correlation (HMQC) spectrum of 0.1 M $[^{6}Li,^{15}N]Et_2NLi$ in 3:2 pentane:toluene with 1 equiv of THF at -118 °C.

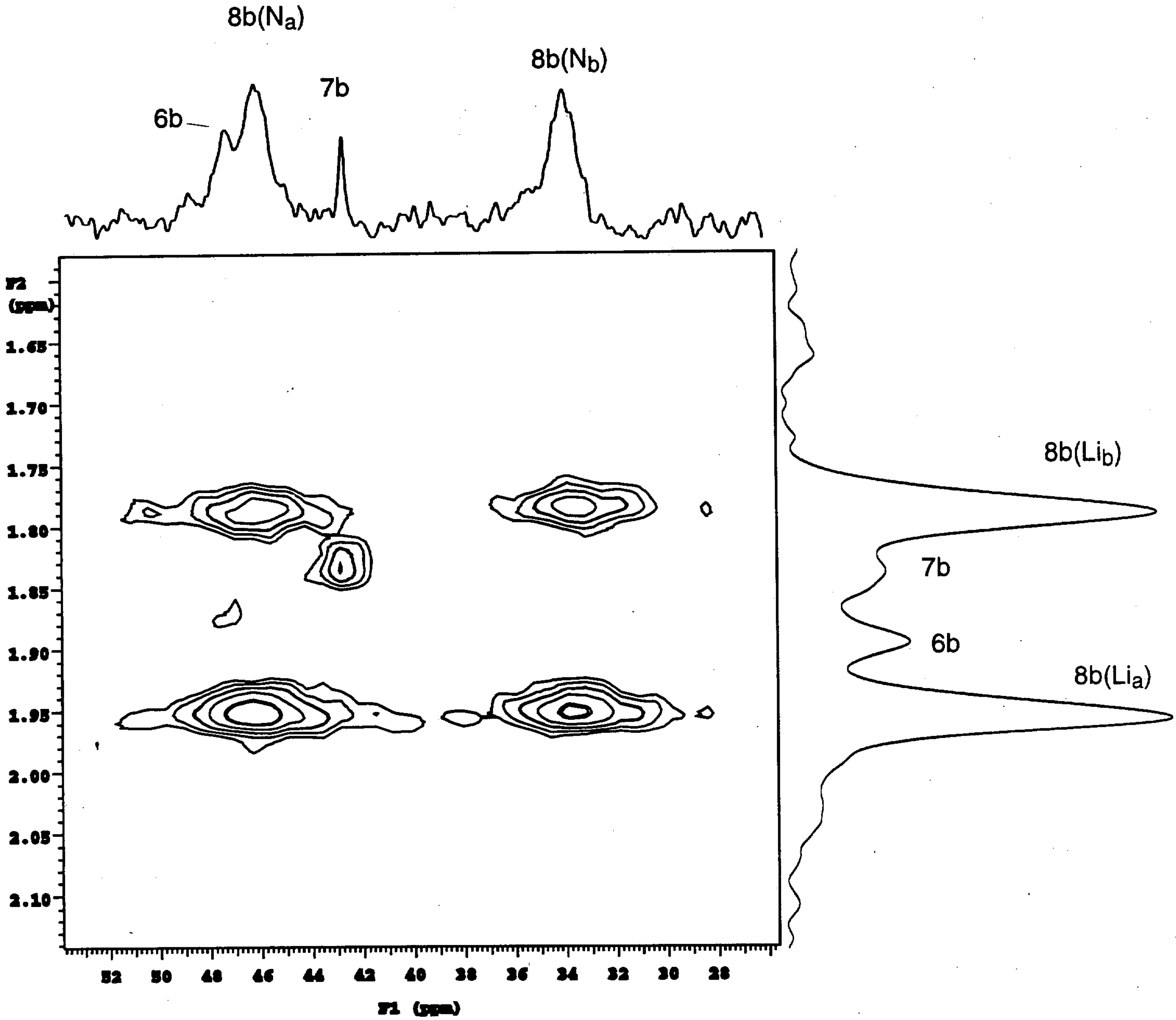

Figure XXVIII. ^{6}Li,^{15}N-heteronuclear multiple quantum correlation (HMQC) spectrum of 0.1 M [^{6}Li,^{15}N]Et_2NLi in 3:2 pentane:toluene with 1 equiv of THF at -87 °C.

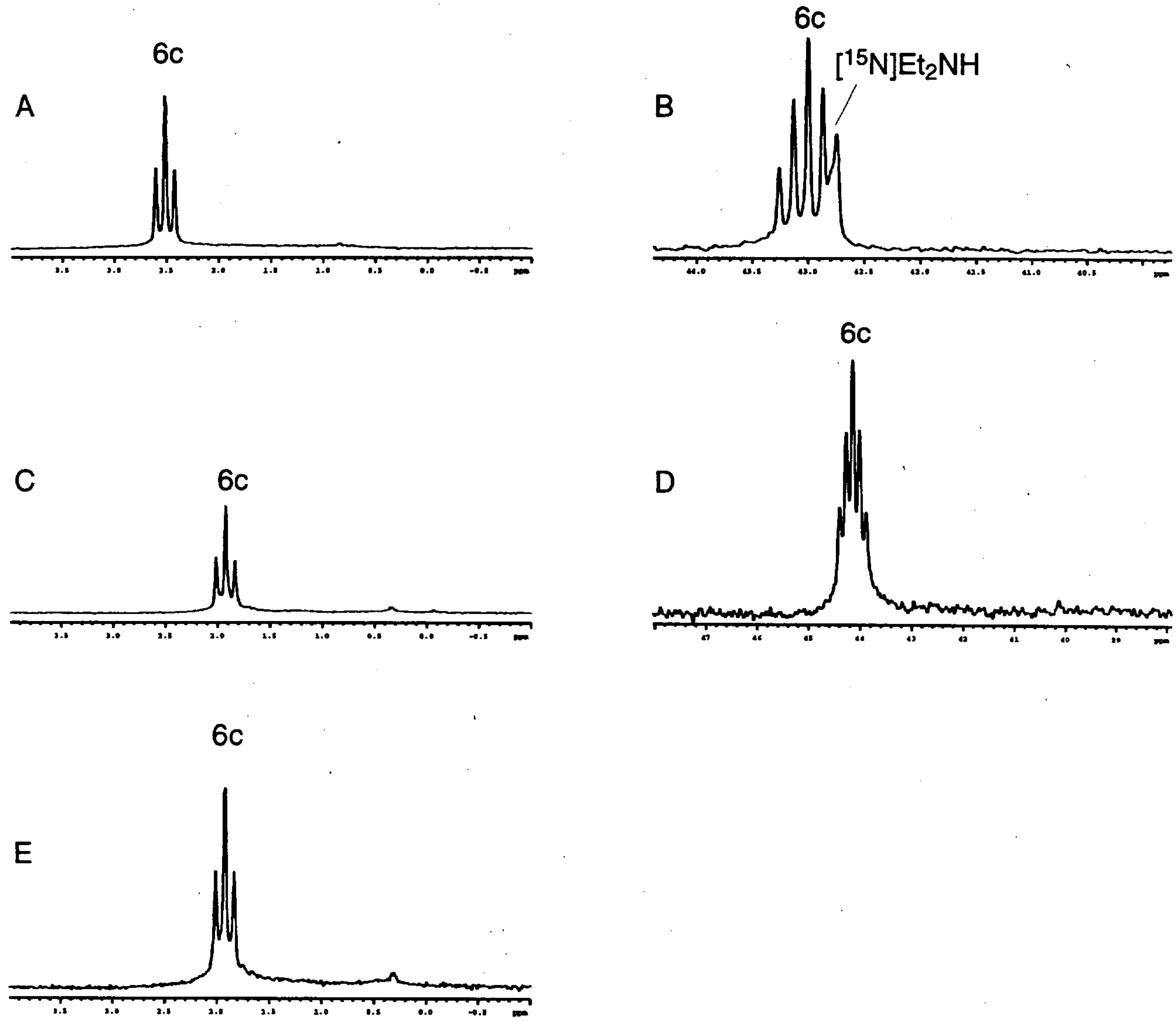

Figure XXIX. ^{6}Li and ^{15}N NMR spectra of 0.1 M [^{6}Li,^{15}N]Et_2NLi in diethyl ether at -100 °C: (A) ^{6}Li NMR spectrum in neat ether; (B) ^{15}N{^{1}H} NMR spectrum in neat ether (Note: Distortion of quintet is caused by presence of [^{15}N]Et_2NH underneath the multiplet.); (C) ^{6}Li NMR spectrum in toluene with 20 equiv ether; (D) ^{15}N{^{1}H} NMR spectrum in toluene with 20 equiv ether; (E) ^{6}Li NMR spectrum in toluene with 5 equiv ether.

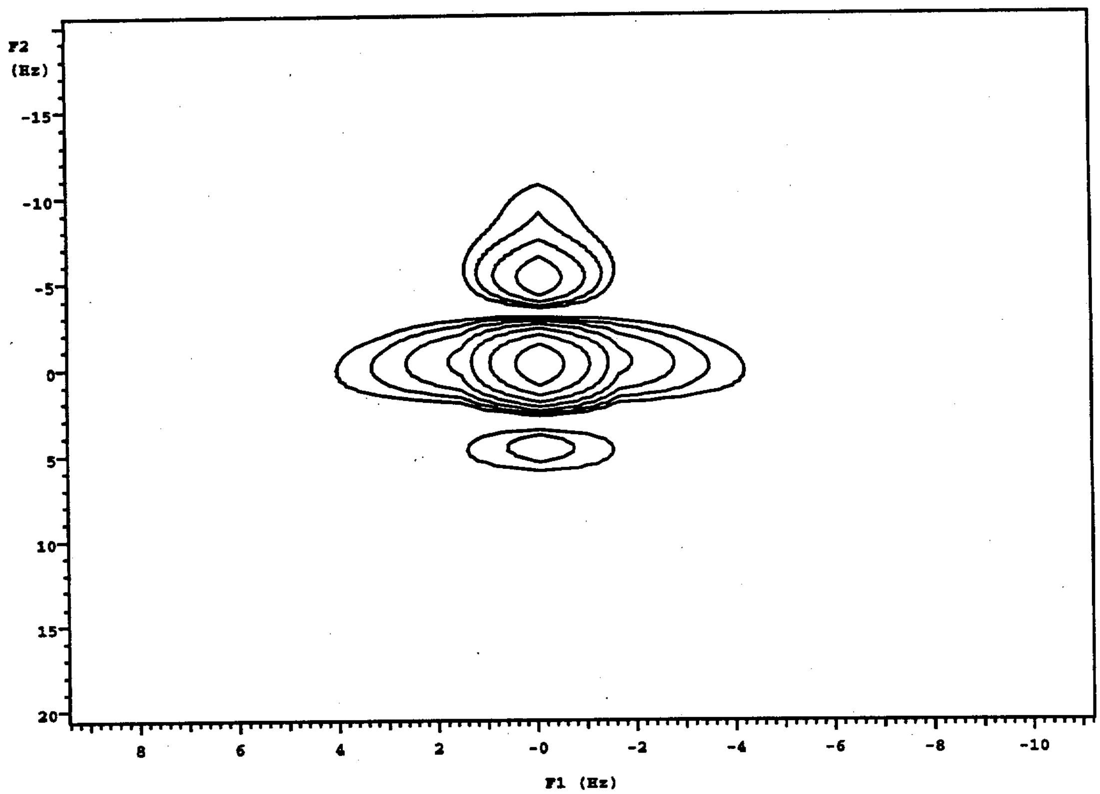

Figure XXX. ^{6}Li-detected ^{15}N zero-quantum NMR spectrum of 0.1 M [^{6}Li,^{15}N]Et_2NLi in neat diethyl ether at -100 °C.