A-C: 6Li, 15N, and 6Li-detected 15N zero quantum NMR spectra for ether-solvated lithiophenylacetonitrile 6 (0.1M). D-F: Corresponding spectra for TMEDA-solvate 7.
G: $^6$Li spectrum of 7 (0.1M). H-I: $^6$Li spectra of 0.1M $[^6]$Li$\text{LiHMDS}$ and 0.1M $[^6]$Li,$^{15}$N$\text{LiHMDS}$ respectively, each containing 0.5 eq. $^{15}$N$\text{phenylacetonitrile}$ and 1 eq. TMEDA. J: $^6$Li spectrum of $[^6]$Li,$^{15}$N$\text{-9}$ (0.05M). K: $^{15}$N spectrum of 7 (0.1M). L: $^{15}$N NMR spectrum of 0.1M $[^6]$Li$\text{LiHMDS}$ containing 0.5 eq. $^{15}$N$\text{phenylacetonitrile}$ and 1 eq. TMEDA.

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M-P: TMEDA and α-cyano carbon region of $^{13}$C NMR spectra of 0.1M $[^6\text{Li},^{14}\text{N}]$-7 at -20, -55, -70, and -90 °C respectively (d$_8$-toluene). Q-R: α-cyano carbon region of $^{13}$C NMR spectra of 0.1M $[^6\text{Li},^{15}\text{N}]$-5 in 1:2 d$_8$-toluene:d$_8$-THF at -90 and -110 °C respectively.
Preparation of $[^{15}\text{N}]$phenylacetonitrile:

A 100 ml 24/40 round-bottom flask equipped with a magnetic stirring bar was charged with $[^{15}\text{N}]$KCN (1.038 g, 15.7 mmol, Cambridge Isotope Lab, dried under vacuum at 50 °C for 45 minutes), 18-crown-6 (4.18 g, 15.8 mmol) and 30 mL of acetonitrile (dried by 3A molecular sieves). Benzyl bromide (7.5 mL, 62.8 mmol) was added by pipet over 5 minutes and the reaction was then put under Argon; a mild exotherm ensued during which nearly all of the solids dissolved. The reaction was monitored by Gas Chromatography (50M Carbowax, 100 °C for 2 minutes, then 10 °C/min for 10 minutes, hold at 200 °C for 2 minutes) by determining the ratio of phenylacetonitrile to remaining benzyl bromide. After 2.5 hours 90% conversion had been achieved, and after 5 hours the reaction was cooled to 0 °C and quenched by the addition of triethylamine (13 mL, 95 mmol). After stirring for 45 minutes at 0 °C and 90 minutes at room temperature, the reaction was poured into 100 mL H$_2$O, and extracted with diethyl ether (1x200 mL, 2x50 mL). The combined ether extracts were washed (2x50 mL 2N HCl, 1x25 mL H$_2$O), dried over MgSO$_4$, filtered, and concentrated in vacuo to give 1.74 g of a yellow oil. Kugelrohr distillation (1mm, 80-100 °C) afforded 1.59 g of a colorless oil (86% yield based on KCN, purity 96 area% by Gas Chromatography).

$^1$H NMR (CDCl$_3$): 3.85 (2H, s), 7.3-7.45 (5H, m).
$^{13}$C NMR (CDCl$_3$): 23.57 (d, $^2$J$_{15\text{N}-13\text{C}}$ = 3.0 Hz), 117.82 (d, $^1$J$_{15\text{N}-13\text{C}}$ = 16.8 Hz), 127.89, 128.02, 129.11, 129.85.
$^{15}$N NMR (1:1 Et$_2$O: Toluene, -90 °C): 247.07 (externally referenced to dimethylethylamine at 25.7 ppm).
IR (NaCl): 2222 cm$^{-1}$ (CN stretch of $[^{14}\text{N}]$phenylacetonitrile occurs at 2255 cm$^{-1}$).