

Preparation of [¹⁵N] HMDS:

1 equiv. ¹⁵NH₄Cl (2.0 g)
3 equiv. TMS-Imidazole (16.5 mL)

¹⁵NH₄Cl was added to a flame dried, Ar-flushed bomb flask with a large stir bar. TMS-Imidazole and about 30 mL freshly distilled dimethylethylamine (DMEA) were added via syringe. The bomb flask was partially evacuated (until the solution starts to boil) and moved to hood, placed in an oil bath at 60 °C behind a blast shield and let stir for 8 days. Be sure the temperature is stable and a blast shield is in front of it.

After 8 days a precipitate should have formed; this might be orange or colorless. The bomb flask was brought to room temperature, and 50 mL freshly distilled pentane were added to the flask via cannula. The contents of the bomb flask were filtered through a Buchner funnel and the filtrate transferred to a 100 mL round-bottom flask. The volatiles were vacuum transferred to another round-bottom flask at -78 °C, monitoring by GC to ensure complete transfer of HMDS (check the vacuum trap for HMDS as well), occasionally warming the remaining filtrate to speed the distillation. When all HMDS had been distilled, the receiving flask was brought to RT.

The volatiles were distilled using a short-path distillation apparatus. First, pentane and DMEA were distilled over at ambient pressure with an oil bath temperature of 50 °C. Again, monitor by GC. HMDS should remain in flask, but occasionally will distill over with DMEA and pentane. Then the bath was cooled to room temperature, and the distillation apparatus was slowly opened to aspirator vacuum, cooling a new receiving flask in an ice bath to collect HMDS.

Romesberg, F. E.; Bernstein, M. P.; Gilchrist, J. H.; Harrison, A. T.; Fuller, D. J.; Collum, D. B. *J. Am. Chem. Soc.* **1993**, *115*, 3475.