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AN EFFICIENT PREPARATION OF POTASSIUM BIS(TRIMETHYLSILYL)AMIDE (KHMDS)

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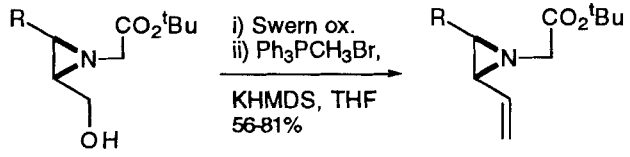
ABSTRACT. A mild and reliable procedure for the preparation of potassium bis(trimethylsilyl)amide (KHMDS) in THF is described.

The introduction of efficient amide bases has played a pivotal role in the development of organic synthesis. Of these, potassium bis(trimethylsilyl)amide (KHMDS)[1] has proven itself to be indispensable in a number of applications, e. g. the Wittig olefination of epoxy aldehydes.[2] In an ongoing investigation of the aza-[2,3]-Wittig rearrangement of vinylaziridines we needed a ready access to the title reagent in order to carry out the transformation outlined in eq 1.[3] Although KHMDS is commercially available we opted to prepare fresh solutions of it in THF, with the hope of increasing the yield of vinylaziridine **1**.



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Initial attempts to prepare KHMDS by adding bis(trimethylsilyl)amine to potassium hydride, pre-washed with hexane, in THF under an inert atmosphere proved to be capricious. After stirring for 24 h at room temperature the heterogeneous slurry was allowed to settle and the supernatant cannulated into a separate flask. Subsequent titration[4] revealed that the base content of the solutions from different runs varied between 1-64% of the theoretical value. In previous studies short induction times (1 h) have been observed in this reaction when using potassium hydride that has been exposed to air repeatedly.[1] However, the induction times recorded in our laboratory in addition to the varying yield obtained made this procedure impractical. Gratifyingly, the encountered problems were overcome by sonicating the mixture of potassium hydride and bis(trimethylsilyl)amine in THF until no further gas evolution could be detected, normally 45 min-3.5 h. This modification of the original procedure has proven to be practical and highly reliable, delivering solutions of KHMDS in THF with a concentration of the reagent that are >80% of the theoretical value.[5]

EXPERIMENTAL

An oven-dried 100 ml round-bottom flask was charged with KH (3.22 g, 20.1 mmol, 25 wt. % in oil) and flushed with argon. The hydride was then washed with hexane (4x5 ml). To the flask was fitted a reflux condenser and then THF (20 ml) and bis(trimethylsilyl)amine (3.81 ml, 18.1 mmol) was added. The resultant slurry was sonicated until the hydrogen gas evolution has ceased and almost all the potassium hydride has been consumed (3.25 h). The slurry was allowed to settle and the supernatant was then cannulated into a Schlenk flask. Titration[4] for the amide base concentration showed it to be 0.80 M, while the concentration of total base was 0.86 M.

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