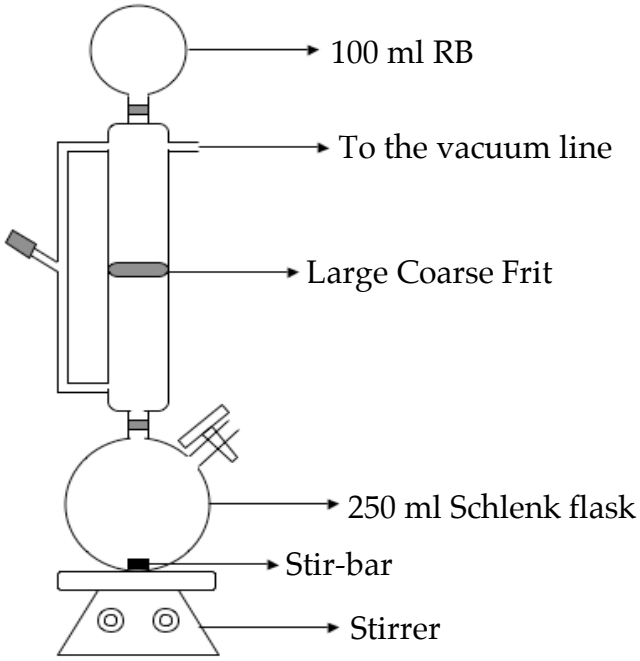
LiHMDS (this is for a large amount of unlabeled base – cut amount of solvent for smaller labeled batches):

1 equiv. *n*-BuLi (ca. 60 mmol)
1.1 equiv. HMDS (14 mL)

100 mL pentane was distilled into a 100 mL round-bottom flask and another 100 mL into a Schlenk flask attached to coarse frit setup.



The Schlenk flask was cooled to 0 °C, and BuLi was added via syringe followed by dropwise addition of HMDS. After stirring 30 min, the solution was warmed to rt and stirred an additional 30 min.

The solvent was removed under vacuum until the solution became cloudy, then warmed in a warm water bath until it dissolves. This was repeated two more times. The flask was then put in a dry ice/acetone bath for 1 hour (do not stir). The bath was removed and the frit flipped to, evaporating all liquid from other side of frit. The solid was dissolved in smallest amount of pentane possible (ca. 20 mL - may need to filter through a fine frit at this point if solution is cloudy). Crystallization/filtration steps were repeated two more times. After final filtration, vacuum was pulled on solid base for about another 30 minutes.

The vacuum line traps were taken down to bring to rt, then emptied and put back up to allow vacuum on base overnight.