

## Preparation of LDA:

Reference:

Reactions:

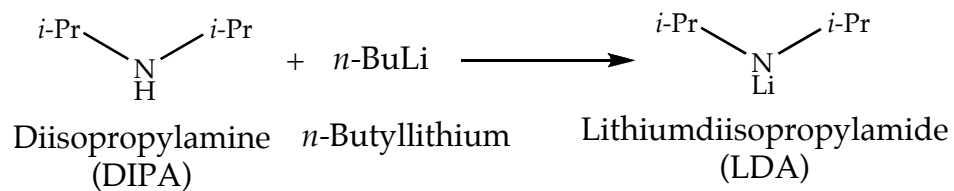
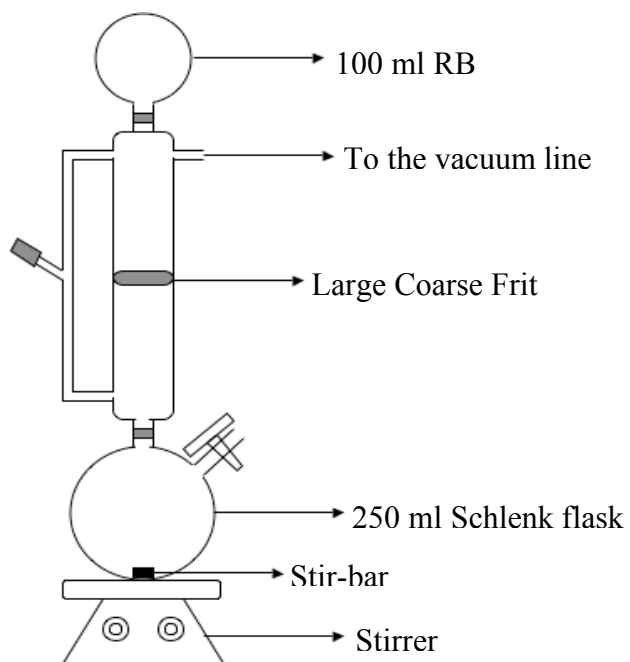


Table:

Reagent	MW (g/mol)	Density (g/ml)	Amount
DIPA	101.19	0.722	12.3 ml (0.088 mol)
n-Butyllithium (1.6 M in Hexanes)	-	-	50 ml (0.08 mol)

Apparatus:



## Procedure:

A large coarse frit was fit with two 250 ml Schlenk flasks (a stir bar at the bottom one). Approximately 50 ml of hexanes was distilled to the frit and ~20 ml of DIPA was distilled to a pear flask. Additional hexanes (~50 ml) was distilled to another pear flask. *n*-BuLi was brought at the room temperature from the freeze. DIPA (~13 ml) was added to the Schlenk flask via syringe at 0 °C with stirring. 50 ml of *n*-BuLi was added to the flask at 0 °C and stirred for 30 minutes (If you are adding a whole bottle, you could use cannula). Ice bath was removed and the reaction was stirred at r.t. for another 30 minutes. Solution turned to a gel (If not, evaporate hexanes till it becomes gel). A warm (??) water bath was put underneath to get the clear solution. A dry ice/acetone bath was kept under the flask so it barely touched the bottom (This is important in order to get good crystal). The flask was allowed to submerge in dry ice/acetone bath very slowly over a long period (1 to 1.5 hr). It was kept submerged till all LDA was settled.

## Recrystallization of LDA:

Around 200 ml of hexanes was distilled to a pear flask. The liquid in the bottom flask was discarded (via syringe) and approximately 150 ml of hexanes was added. The solution was stirred at 65 °C under Ar to dissolve LDA (for a period of over 2 hr). Once all the LDA was dissolved, it was allowed to come to r.t. Stirring was stopped; dry ice/acetone bath was kept under the flask barely touching the bottom. The flask was allowed to submerge in dry ice/acetone bath very slowly over a long period (1 to 1.5 hr). It was kept submerged till all LDA was settled (it should be white, if not recrystallize again). It was kept under Ar overnight at r.t.

Next day, the liquid was discarded. The frit was flipped and the solvent was evaporated. 10 ml of freshly distilled hexanes was added to LDA crystal to wash and the liquid was discarded. Washing was repeated twice. After removing the solvent, the whole system was put under full vacuum for 3-4 hrs. the entire frit was moved to glass box and white crystalline LDA was transferred to a different flask.