LDA preparation from DIPA and Li metal

	Molecular weight	mmol	Quantity	Equiv
Isoprene	68.12 g/mol (0.681 g/ml)	25.0	2.27 ml	0.5
$^{6}Li^{0}$	6.0 g/mol	50.0	0.30 g	1.0
^{15}N-DIPA·HCl	138.65 g/mol	50.0	6.30 g	1.0
DMEA	73.14 g/mol (0.675 g/ml)	/	40 ml	/
n-BuLi	2.5 M in hexanes		18.2	

Materials:

Isoprene: purchased from Aldrich, 99% purity, inhibited with catechol distilled from molecular sieves.

$^{6}Li^{0}$: isotopically enriched lithium (group stock) - stored in glove box.

DIPA·HCl: Diisopropylamine hydrochloride was prepared from $^{15}NH_4Cl$ as described elsewhere.

DMEA: Dimethylethylamine was purchased from Aldrich and distilled and dried over sodium/benzophenone as a purple solution.

***n*-BuLi:** purhased as 2.5 M n-BuLi solution in hexanes.

Procedure:

n-BuLi (2.5M, ~18.2 ml) was added to a suspension of DIPA·HCl (6.30g) and 1-2 mg of 1,10 phenanthroline in 30 ml DMEA. The endpoint of the titration and coincident liberation of the amine is achieved when the solution turns from pale-yellow to a permanent rust-red color. Patience is required at the last 10% of the titration (sometimes minutes are required for suspension to turn back to pale yellow color). Rapid stirring in a 250 ml RB will speed up this process. Cooling is necessary in the beginning due to the exothermicity of the titration.

Transfer all liquids via direct distillation to a 100 ml RB with sidearm, which are subsequently added to ^{6}Li metal (0.30 g) in a 250 ml RB swivelfrit assembly and a receiving 250 ml Schlenk flask.

Distilled isoprene (2.27 ml) was disolved in 10 ml dry DMEA and added over 30 minutes at room temperature via syringe pump to the solution of lithium, diisopropylamine and DMEA. Keep the reaction in a room temperature water bath to avoid exothermicity and temperature above 35 ºC.

After three to four hours of reaction time, the mixture was filtered through a fine frit and evaporated to dryness. The solid LDA was transferred to the glovebox for subsequent recyrstallization.