

### Preparation of *n*-BuLi (<sup>6</sup>Li and <sup>7</sup>Li)

150 mL hexanes was distilled into a 250 mL 3-neck round-bottom flask attached to a large fine frit (be sure everything is flame dried). <sup>6</sup>Li (2.0 g Li, 0.33 mol) was cut into small pieces as well as 1 cube of sodium. A stopper on the 3-neck RB was briefly replaced with a solid addition funnel, and the metals were added under Ar flow, quickly replacing the stopper. The mixture was sonicated for 30 min, cooled to 0 °C, and 1-chlorobutane (18 mL, 0.17 mol) was added over 4 hours using syringe pump. The mixture was then stirred overnight, plus full next day (40 h).

The following procedure applies to labeled (<sup>6</sup>Li) as well as to the purification of unlabeled (<sup>7</sup>Li) *n*-BuLi:

On third day, BuLi was slowly filtered through the fine frit, rinsing the solids with freshly distilled hexanes (4x10 mL). The liquid was transferred via cannula to a flame dried 250 mL RBF with T-joint adapter. Hexanes were removed under vacuum, leaving neat BuLi in the flask.

About 15 mL freshly distilled pentane was carefully added to the neat BuLi. Measure about 50 mg diphenylacetic acid into each of 3 vials and dissolve in 1 mL freshly distilled THF. The BuLi solution was titrated to 3.0-3.3 M (add or remove pentane as needed). The flask was put UNDER VACUUM and placed in liquid nitrogen to shock freeze for about 2 minutes, then immediately moved into a -95 °C freezer. Crystals may take several days to appear. If no crystals appear after two days, shockfreeze for a bit longer and try again.

The crystals were warmed to RT, and pentane was carefully added to the desired molarity (about 1.6 M – check by titrating against diphenylacetic acid using same procedure as before). Store in freezer.