

## Procedures for Synthesis of $\text{tBuSn}(\text{NEt}_2)_3$

(to be performed by fully trained personnel with appropriate equipment)

### Procedure 1:

**Step 1.** Place  $\text{Sn}(\text{NEt}_2)_4$  (4.08 g, 10.0 mmol) into an appropriate reaction vessel for air-sensitive chemistry, such as a Schlenk flask, under inert atmosphere (e.g., dry nitrogen or argon), and dilute with 50 mL of  $\text{Et}_2\text{O}$ .

- Use a magnetic stir bar or similar means to provide agitation/stir

**Step 2.** Prepare 6.2 ml of a 1.6 M solution of t-BuLi (i.e., 10 mmol of t-BuLi) in an appropriate solvent (n-pentane, or similar non-polar solvent) under inert atmosphere. (**Caution: t-BuLi is very pyrophoric so extreme caution must be used**)

**Step 3.** Place the t-BuLi solution in an appropriate vessel, such as an addition funnel, that is connected to the reaction vessel, with both under inert atmosphere.

**Step 4.** Cool the reaction vessel by using a dry ice bath ( $-78\text{ }^\circ\text{C}$ ).

**Step 5.** Add the t-BuLi solution dropwise into the reaction vessel with stirring.

**Step 6.** After the addition, allow the reaction mixture to return to room temperature by removing the dry ice bath; the attached vessel for adding the t-BuLi solution may be removed at this time.

**Step 7.** Stir the reaction mixture for 2 hours at room temperature, under inert atmosphere.

**Step 8.** Remove the solvent/volatile components under reduced pressure.

**Step 9.** Extract the resulting product with benzene (e.g., 30 mL) or other non-polar solvent; filter the mixture to remove the precipitate.

**Step 10.** Remove the volatile components from the filtrate of Step 9 under reduced pressure to give the final product.

**Step 11.** Collect  $^1\text{H}$ ,  $^{13}\text{C}$  and  $^{119}\text{Sn}$  NMR spectra from a sample of the final product.

**Note:** the reactants and the products should be maintained under inert atmosphere or vacuum at all times to prevent any possible decomposition by the ambient environment. They should also be protected from ambient light to prevent any possible decomposition from prolonged exposure.

### Procedure 2:

Conduct the same steps as in procedure 1 with the following modification:

Instead of employing 10 mmol of t-BuLi, use an excess amount of t-BuLi (e.g., by 20%) in Step 2.