

Procedures for Synthesis of $\text{iPrSn}(\text{NMe}_2)_3$

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

Step 1. Place LiNMe_2 (81.75 g, 1.6 mol) into a reaction vessel that is appropriate for air-sensitive chemistry, such as a Schlenk flask, under inert atmosphere (e.g., dry nitrogen or argon), and mix with 700 mL of anhydrous hexanes (or similar solvent)

- Use a magnetic stir bar or similar means to provide agitation/stir.
- Note: The amount of LiNMe_2 and solvent may be scaled proportionally for the size of reaction vessel chosen

Step 2. Prepare iPrSnCl_3 (134.3 g, 0.5 mol) under inert atmosphere.

- The amount of iPrSnCl_3 may be scaled proportionally based on the size of reaction vessel chosen.
- Note: the molar ratio between LiNMe_2 and iPrSnCl_3 should be maintained to be approximately 3.2:1.0.

Step 3. Place iPrSnCl_3 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 iPrSnCl_3 dropwise into the reaction vessel, over a period of 2 hours or scaled properly according to the amount chosen.

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 iPrSnCl_3 may be removed at this time.

Step 7. Stir the reaction mixture overnight at room temperature, under inert atmosphere.

Step 8. Allow the reaction mixture to settle and then filter the reaction mixture to remove solid precipitates.

Step 9. Remove the solvent/volatile components from the filtrate under reduced pressure.

Step 10. Distill the residue obtained from Step 9 under reduced pressure.

- Example distillation condition: pressure ca. 1.4 Torr, temperature $50\text{--}52\text{ }^\circ\text{C}$.
- Depending on availability of equipment, comparable distillation conditions may also be employed.

Step 11. Collect ^1H , ^{13}C and ^{119}Sn NMR spectra from a sample of the distillate.

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.