

Experimental Procedure – Preparation of N-tert-Butoxycarbonyl L-Alanine

1. Place 0.90 g of L-alanine in a 50-mL round-bottom flask containing a stirbar.
2. Add 5 mL *tert*-butanol and 5 mL 3 M NaOH.
3. Using the 5-mL syringe, transfer 2.5 mL of di-*tert*-butyl dicarbonate to the round-bottom flask and continue stirring for 45 min at room temperature.
4. Add 10 mL of water to the reaction mixture and transfer the solution to a separatory funnel. Rinse the round-bottom flask with 1–2 mL of water and transfer the rinse to the separatory funnel.
5. Add 25 mL of diethyl ether to the separatory funnel and shake the funnel gently, venting as necessary. Separate the layers and acidify the aqueous layer to pH 2 with 3 M HCl.
6. Extract the aqueous layer with two 10-mL portions of diethyl ether and then wash the combined organic layers with 10 mL of brine. Dry the organic layer over several spatula-tips full of anhydrous sodium sulfate and remove the solid by gravity filtration with a glass funnel.
7. Concentrate the solution to between 4-5 mL. If the solution is a little cloudy, add a spatula tip of sodium sulfate and decant into a new flask.
8. Add approximately 25 mL of hexanes to the ethereal solution, stir the solution with a glass stirring rod, and place the flask in an ice-water bath for 10 min. If no solid precipitates, scrape the bottom of the flask with a spatula until precipitate starts to form.
9. Weigh the recrystallized product, determine its melting point, and obtain an IR spectrum.