

## **Experimental Procedure – Preparation of Methyl L-Phenylalaninate Hydrochloride**

- 1.** Place 1.0 g of L-phenylalanine in a 25-mL round-bottom flask containing a one-stirbar, add 5 mL of MeOH, and begin stirring the mixture.
- 2.** Add 0.5 mL of thionyl chloride dropwise using a syringe. Equip the round-bottom flask with a reflux condenser and heat the mixture, which should be homogeneous under gentle reflux for 45 min.
- 3.** Allow the reaction mixture to cool to room temperature and then transfer the solution to a 50-mL Erlenmeyer flask. Rinse the round-bottom flask with a 0.5–1-mL portion of methanol and transfer this rinse to the Erlenmeyer flask.
- 4.** Place the Erlenmeyer flask in an ice-water bath and add 25 mL of diethyl ether. Using a glass stirring rod, scratch the inside of the flask at the air-liquid interface to induce crystallization.
- 5.** Collect the white solid by vacuum filtration and air-dry it.
- 6.** Recrystallize the crude product by a mixed solvent recrystallization with ethyl acetate and hexanes. Add 0.5 mL methanol to the flask (if it does not dissolve, add additional methanol in 0.1 mL increments until it is dissolved). Place on a hot plate that is set to 120. Start adding diethyl ether 0.5 mL at a time until the solution becomes cloudy. Remove from the hot plate and allow to cool to room temperature.
- 7.** Collect the white solid by vacuum filtration and air-dry it.
- 8.** Weigh the recrystallized product, determine its melting point, and obtain an IR spectrum.