

### Procedure:

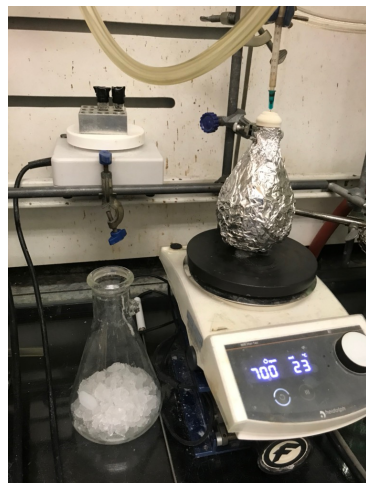
In a round bottom flask protected from light,  $\text{PBr}_3$  (0.51 mL, 1.0 equiv, 5.4 mmol) was added dropwise to a solution of allylic alcohol (1.0 g, 1.0 equiv, 5.4 mmol) in anhydrous diethyl ether (0.2 M, 27 mL) at 0 °C. The reaction was stirred at 0 °C for 1 h, then warmed to 23 °C and stirred overnight. After 12 h, the crude mixture was poured onto ice in the dark and then transferred to a separatory funnel. The organic layer was collected, then the aqueous layer was extracted with pentanes (2x). The combined organic layers were washed with saturated aqueous sodium bicarbonate (3x) and brine (1x), then dried over magnesium sulfate and filtered. The solution was concentrated to ~1 M (rotovap at 20 °C, >200 mbar) and yield was determined by NMR.

### Step 1:



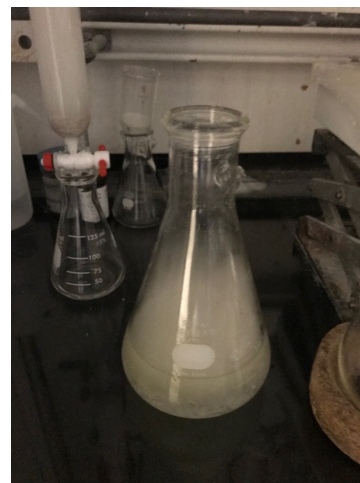
Acquire necessary PPE:  
**chemical resistant gloves**, nitrile gloves, lab coat, and safety glasses.

### Step 2:



Perform the reaction in a round bottom flask protected from light. After stirring overnight, prepare an Erlenmeyer flask with ice.

### Step 3:



In a dark fume hood, **cautiously** pour the crude reaction over ice. Mix gently and allow ice to melt.

### Step 4:



Transfer to a separatory funnel. Collect the organic layer, then extract the aqueous layer with pentanes (2x). Wash combined organic layers with aq.  $\text{NaHCO}_3$  and brine.



### Step 5:



Concentrate under reduced pressure (20 °C, >200 mbar) using a rotovap vented into a fume hood with closed sashes.

### Step 6:



Remove from rotovap when concentration is ~ 1 M. Determine yield by NMR.

### Step 7:



Protect from light and temporarily store under N<sub>2</sub> in a freezer while preparing your enolate.

### Step 8:

Rinse all glassware with aq. ammonium hydroxide and allow to quench in a fume hood overnight.



### Step 9:

Dry the solution of allylic bromide in pentanes with magnesium sulfate, then add dropwise to the enolate via cannula while protecting from light.

