

## Procedure

1. Weigh out the appropriate amounts of the compounds **(1)** & **(2)**, using an analytical balance. To calculate the amount of material required, use the spreadsheets incorporated into the word documents found at the top of the page. After determining how much material is required for the reaction, transfer compounds **(1)**, the  $\text{CH}_2\text{Cl}_2$  (50 mL) and pyridine (120mL) to a round bottomed flask. Place a support stand with a clamp next to the stirplate. If available, put a cork ring on the stir plate
2. Place the flask on the stir plate and clamp. Add a magnetic stir bar and begin stirring
3. Add **(2)** to the flask drop wise at room temperature over the course of 20 to 30 minutes
4. Stir the mixture at room temperature for 24 hours. Reaction progress may be monitored by TLC (n-hexanes : ethyl acetate 9:1)
5. When the reaction is complete, quench the reaction by adding 200 mL of distilled  $\text{H}_2\text{O}$ , and then stir for additional 30 minute
6. Next, the water needs to be removed using a separatory funnel. Pour the reaction mixture into the separatory funnel, the aqueous phase will be on the top this time if  $\text{CH}_2\text{Cl}_2$  is used as the solvent. Cap and gently shake the funnel for about a minute to mix the contents. Place the flask in a stand and let sit for at least 10 minutes to allow the organic phase to separate from aqueous phase. The product will remain in the organic phase. Collect the aqueous phase and the organic phase in two separate flasks or beakers. Retain the organic phase as it contains the product.
7. Residual product may still be in the aqueous phase. To recover any product in the aqueous phase, wash the water with ~ 40 mL of THF again using the separatory funnel. Please be careful to remember that the water will now be the bottom layer, and the THF will be the top layer inside the funnel. Combine the THF fraction with the organic solution from the previous step 5. Continue washing until all of the product is removed from aqueous phase. TLC can be used to see if any residual product The location of the product may be monitored by TLC.
8. To remove any residual water from the organic phase, the organic phase is dried of  $\text{MgSO}_4$ . To do this, add a couple of grams of  $\text{MgSO}_4$  to the organic phase. This will trap any water in solution. If the  $\text{MgSO}_4$  clumps together, it has trapped water, and there may still be water in organic phase. However, if the  $\text{MgSO}_4$  appears like a powder, floating in solution, then all of the is trapped.
9. The  $\text{MgSO}_4$  is removed using by bay passing the organic phase through filter paper or a Buchner funnel step
10. The organic solvent is removed under vacuum, yielding an oil that requires no further purification

