

Fischer Esterification Procedure

1. Place 25 mL of distilled water in a 150 mL beaker into an ice-water bath. Also place into the ice water bath one mL of 6 M NaOH in a test tube, and 10 mL of saturated sodium bicarbonate in a test tube
2. Add 15 mL of the 1 M benzoic acid in methanol solution to the 50 mL round bottom flask along with the rice stir bar.
3. Add 0.5 mL of concentrated sulfuric acid to the round bottom flask and attach the west condenser. Attach the water lines for refluxing.
4. Heat the round bottom flask in the heating block and set the hotplate to $\sim 220^{\circ}\text{C}$.
5. After 30 minutes the reaction is complete. **Don't start the timer until condensation is observed in the condenser.**
6. **Carefully** remove the round bottom flask from the hotplate and pour the reaction mixture into a 150 mL beaker that contains the 25 mL of water.
7. Cool the solution in an ice-water bath for 5 minutes to ensure that the solution is below room temperature (it doesn't have to be especially cold, just below room temperature)
8. Add 1mL of 6M NaOH to the cooled solution then add roughly half of the saturated sodium bicarbonate and test the pH. Continue adding sodium bicarbonate 1mL at a time until the pH reaches 7. Transfer this neutralized solution to your separatory funnel.
9. Add 15 mL of diethyl ether to your separatory funnel and then extract the organic phase. Drain the aqueous layer and set aside.
10. Wash the organic layer with 15 mL of saturated sodium chloride solution. Isolate the organic layer. **DO NOT discard anything until analysis of your product has been completed.**
11. Add sodium sulfate to dry the product and decant the solution into a new beaker. Shoot 1 μL of this sample on the GC and identify the ester by comparing the retention time to the standard which can be found on the course webpage.
12. To isolate the pure product, remove the ether by short path distillation. Pour the solution into a 25 mL round bottom flask along with the rice stir bar. Connect the flask to the short path vacuum adapter. Use a 50 mL round bottom flask as your receiving flask that is submerged in an ice water bath. Be sure to have Keck clamps attached to both round bottom flasks. Set the hotplate to 100-150 and the stirring to 4-5. Continue removing the ether until it stops boiling.
13. Isolate the ester and determine the percent yield, take an IR and NMR (NMR is 128L only).