

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 ~220 °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 the 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 of anything until analysis of your product has been completed.**
11. Add several spatula tips of sodium sulfate to the product to dry it and decant the solution into a new beaker. Shoot 1 μ L of this sample on the GC.
12. To isolate the pure product do one of the following.
 - a) If you are in 128L, remove the diethyl ether by evaporating it using the air hose.
 - b) If you are in 220C, remove the diethyl ether by simple distillation. Use the 25 mL round bottom flask as the distillation flask with a stirbar. Use the 50 mL round bottom flask as your receiving flask and submerge it in ice water. Set the hotplate to about 150 °C and the stirplate to between 4 and 5.
13. Isolate the ester and determine the percent yield, take an IR and NMR (NMR is 128L only).