

Fischer Esterification Procedure

1. 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.
2. Add 0.5 mL of concentrated sulfuric acid to the round bottom flask and attach the west condenser. Attach the water lines for refluxing.
3. Heat the round bottom flask in the heating block and set the hotplate to ~220 °C.
4. After about 30 minutes the reaction is complete. **Don't start the timer until condensation is observed about a quarter of the way up the condenser.**
5. **Carefully** remove the round bottom flask from the hotplate and pour the reaction mixture into a 150 mL beaker that contains ~25 mL of water.
6. Cool the solution in an ice-water bath to ensure that the solution is below room temperature (it doesn't have to be especially cold, just below room temperature)
7. Add 10% aqueous sodium bicarbonate to your cooled solution until the pH is around 7. This typically requires around 60 mL of sodium carbonate but add the sodium carbonate slowly and test the pH occasionally to verify that the pH is increasing. Transfer this neutralized solution to your separatory funnel.
8. Add 15 mL of diethyl ether to your separatory funnel and then extract the organic phase. Drain the aqueous layer and set aside.
9. Wash the organic layer with 15 mL of saturated sodium chloride solution. Isolate the organic layer. The aqueous layers can be poured down the sink but **DO NOT discard of anything until analysis of your product has been completed.**
10. Add several spatula tips of sodium sulfate to the product to dry it and then 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.
11. To isolate the pure product carefully decant or filter the liquid so that no sodium sulfate remains and then follow the directions of your TA to remove the left-over diethyl ether. Once pure product has been isolated take an IR and NMR (NMR is 128L only).