

## Procedure

1. Add 3 mL of the 1 M benzoic acid in methanol solution to the 5 mL conical vial along with the rice stir bar.
2. Add ~4 drops of sulfuric acid to the conical vial and attach the small condenser. Attach the water hoses to the condenser using metal hose clamps. Turn the water on so that it is running slowly.
3. Heat the conical vial in the heating block (set the hotplate to ~200 °C).
4. After about 30 minutes the reaction is complete. **Don't start the timer until condensation is observed in the condenser.**
5. **Carefully** remove the conical vial from the hotplate and pour the reaction mixture into a 25 mL beaker that contains ~5 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 carbonate to your cooled solution until the pH is around 8. Add it slowly because bubbles of CO<sub>2</sub> gas will be released. This typically requires around 10 mL of sodium carbonate but add the sodium carbonate slowly and test the pH occasionally to verify that the pH is increasing.
8. Once the solution is at pH 8, transfer it to your separatory funnel. Add 3 mL of diethyl ether and shake/vent for about 10 minutes. Drain the aqueous layer (the bottom layer) into a beaker and set the beaker aside. Add 5 mL of saturated sodium chloride to the separatory funnel and shake/vent for 10 minutes.
9. Drain the aqueous layer (the bottom layer) into a beaker and set it aside. Now drain the ether into a clean beaker.
10. Add sodium sulfate to the beaker to dry it. Filter the sodium sulfate using your glass funnel and a piece of cotton. Collect the ether into another clean flask.
11. Obtain an IR of the ester, obtain a GC chromatogram and a proton NMR spectra.

## Hazard Analysis for Fischer Esterification

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Hazard	Scenario
1. Sulfuric acid	Drop of acid on gloves, and your head itches.
2. Heating reaction in cracked rb flask	Removes goggles to wipe sweat from forehead next to hotplate.
3. Beaker of ethanol on edge of bench	Walking in lab with lab coat unbuttoned.
4. Reaction in hood	Sash pushed all the way up.

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## Data Analysis Questions for Fischer Esterification (There is no percent yield calculation)

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### A. GC Chromatogram

- What is the retention time of your product?
- What is the standard retention time for methyl benzoate?
- Do the two match up?
  - Are the two retention times slightly different?
  - What sources of error are there?
- Is there something odd about the peak shape?
  - What could be causing this shape?
  - Does this shape cause any issues with your retention time?

### B. IR

- IR functional group region analysis
  - What peaks are present that should be present?
  - What peaks are absent that should be absent?
  - Are there any peaks present or absent that should not be?
  - What conclusion can you draw about the functional group region?
- IR fingerprint region analysis
  - What peaks are present that should be present?
  - What peaks are absent that should be absent?
  - Are there any peaks present or absent that should not be?

- iv. What conclusion can you draw about the fingerprint region?
  - c. IR final analysis
    - i. Does the IR support the synthesis of methyl benzoate?
    - ii. Why or why not?
  - C. NMR (For 128L Only)
    - a. Is the correct number of peaks present?
      - i. If there are any missing, which ones?
      - ii. Is the chemical shift correct for each peak?
      - iii. Is the splitting pattern for each peak correct?
      - iv. Is the integration of each peak correct?
    - b. Are there any peaks absent? If yes, which ones?
    - c. Are there any peaks present that should not be?
      - i. How many?
      - ii. What are the chemical shifts, splitting pattern and integration?
      - iii. What is the potential source of these peaks?
    - d. NMR final analysis
      - i. Does the proton NMR support the synthesis of methyl benzoate?
      - ii. Why or why not?
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