

- Place ~ 0.10 g benzil into a
3 ml conical vial w/
Spin vane & Air Condenser

- Add 0.30 mL 95% EtOH

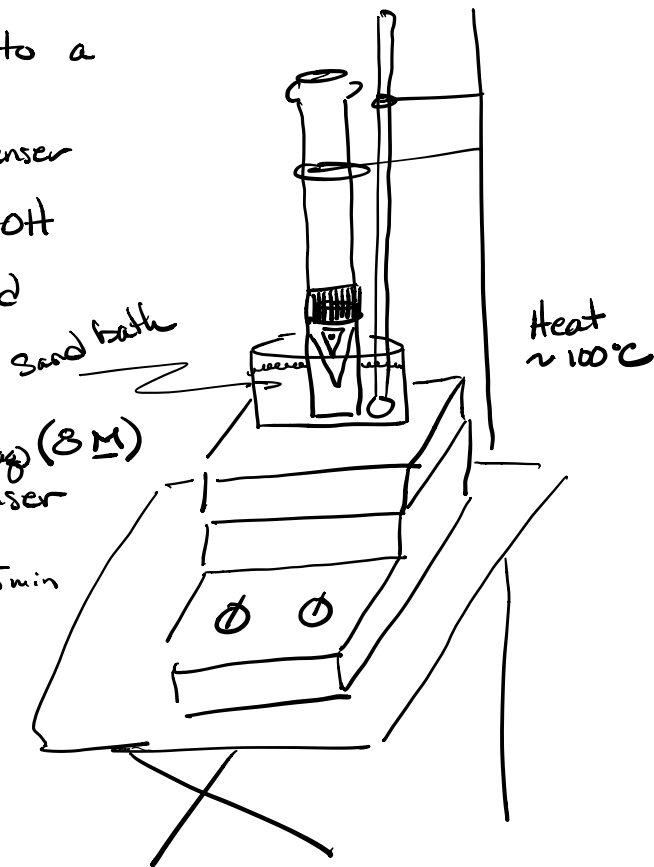
- Heat $\sim 100^\circ\text{C}$ until solid
dissolves

- Add 0.25 mL of KOH (aq) (8 M)
dropwise through Condenser

- Boil w/ stirring for ~ 15 min

- Turns Blue/Black

- Cool to room temp



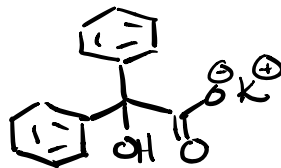
work up

- Transfer contents to 10 mL beaker

- Ice bath for 15 min \rightarrow may need to use scratching
Rod to induce crystallization.

- Hirsch funnel & Rinse w/ 3 x 1 mL 95% EtOH 0°C

\Rightarrow potassium Benzilate

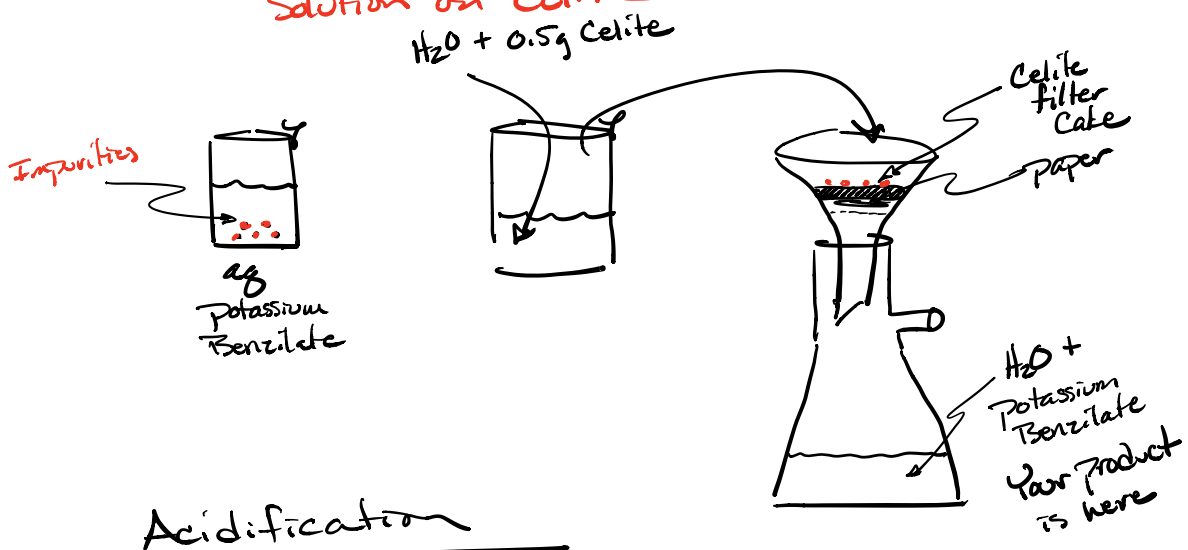


Highly water soluble
 \Rightarrow Do not Rinse w/ H_2O

- Transfer Solid to Small 10ml Erlenmeyer Flask with 3 ml Hot H_2O .
- Stir until the potassium benzoate dissolves

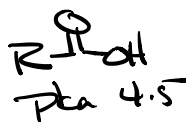
⇒ Check to see if there is any solid present.
 The solid should all dissolve
 Anything that doesn't dissolve is not your product

⇒ if there are solids then we filter the solution on Celite

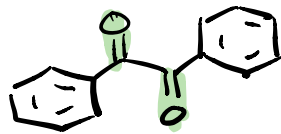


Acidification

- Add 0.5 ml 1M HCl w/ stirring
- Take pH down to ~ 2
- Benzoic acid should precipitate out
- Cool & Ice for 5-10 min
- filter on Hirsch
- mass
- mp
- FTIR

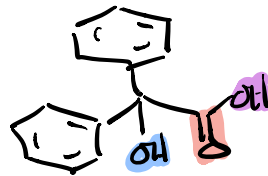


mp



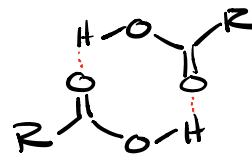
dipole-dipole

95°C

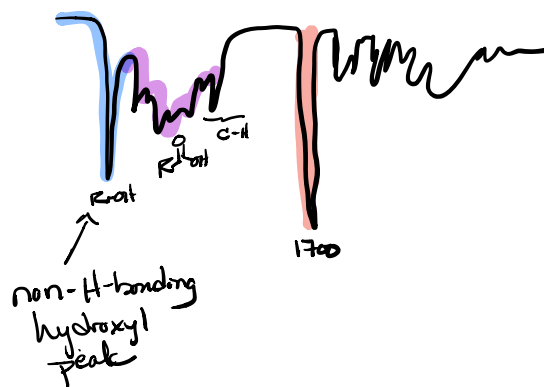
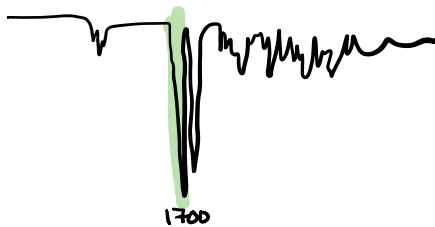


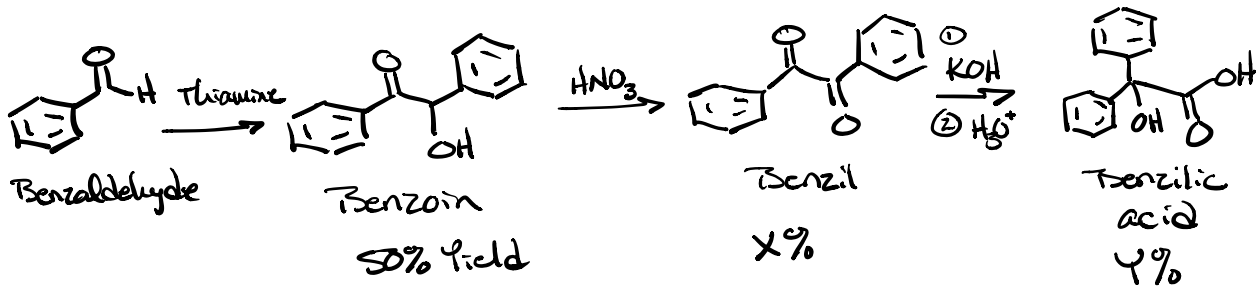
H-bonding

150°C



Dimer





Each step has own yield
overall yield

$$\frac{50\%}{100} \times \frac{X\%}{100} \times \frac{Y\%}{100} \times 100 = \text{overall isolated Yield}$$

Typical falls ~5-3%

Solid Phase Synthesis

