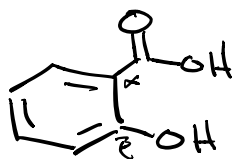


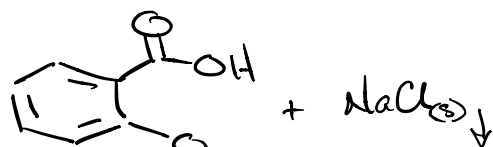
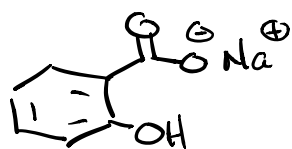
Synthesis of Acetylsalicylic Acid



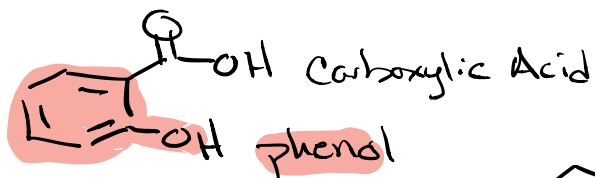
(Salix latin for willow tree)

Salicylic Acid
o-hydroxy acid

1853 Charles Gerhardt



Acetylsalicylic Acid

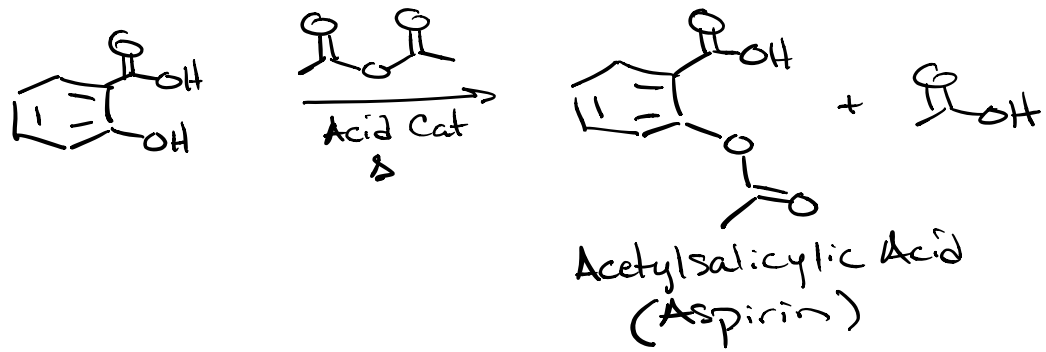


OH pKa 15.7

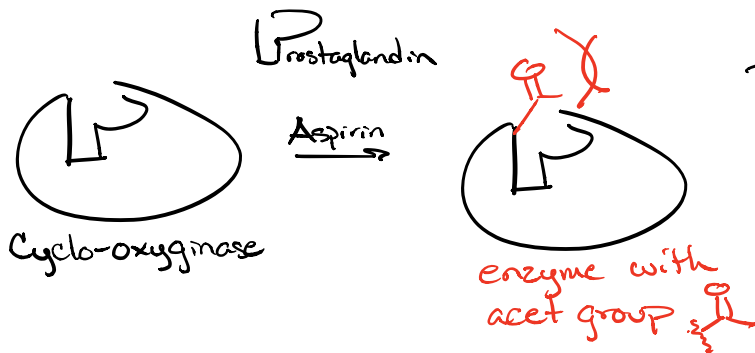
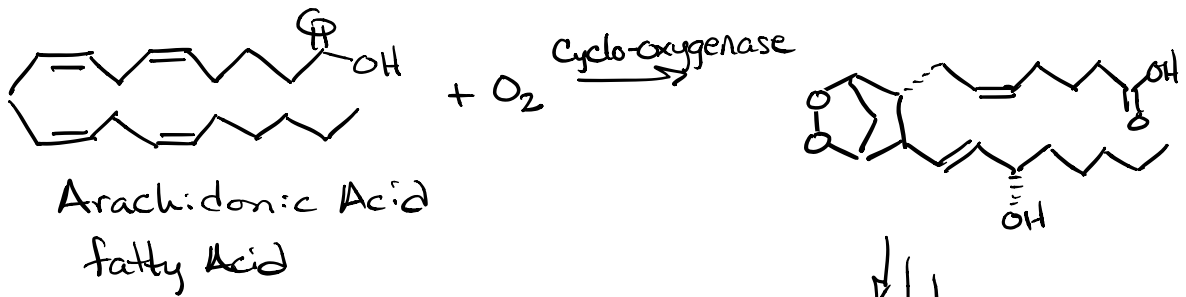
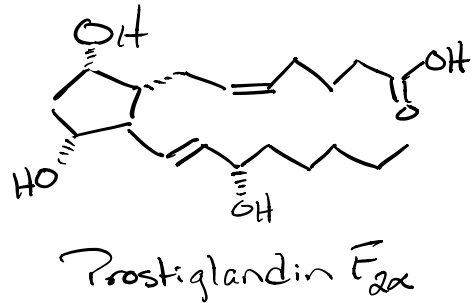
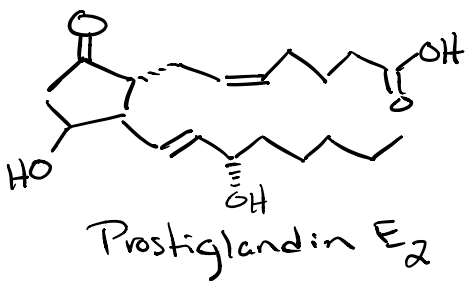
OH pKa 9

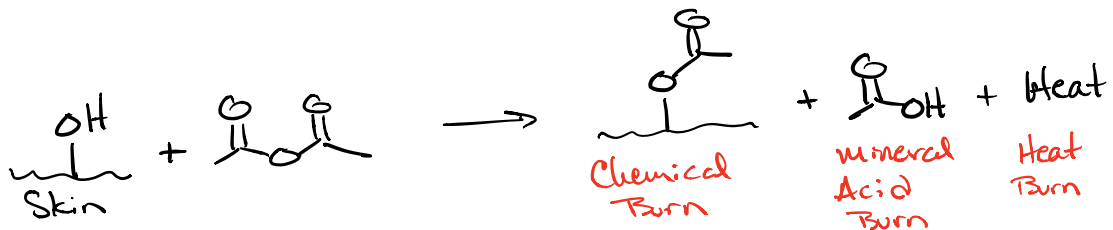
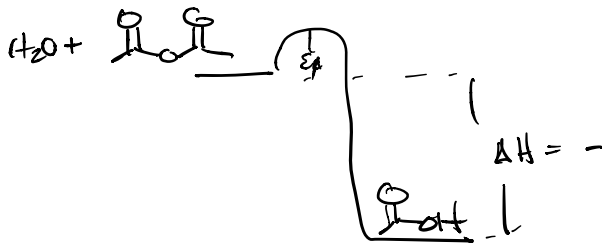
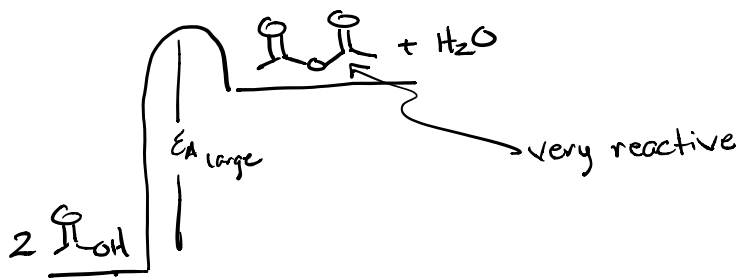
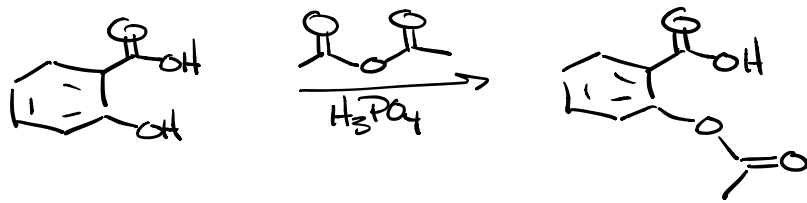
OH pKa 4.5

1897 Bayer

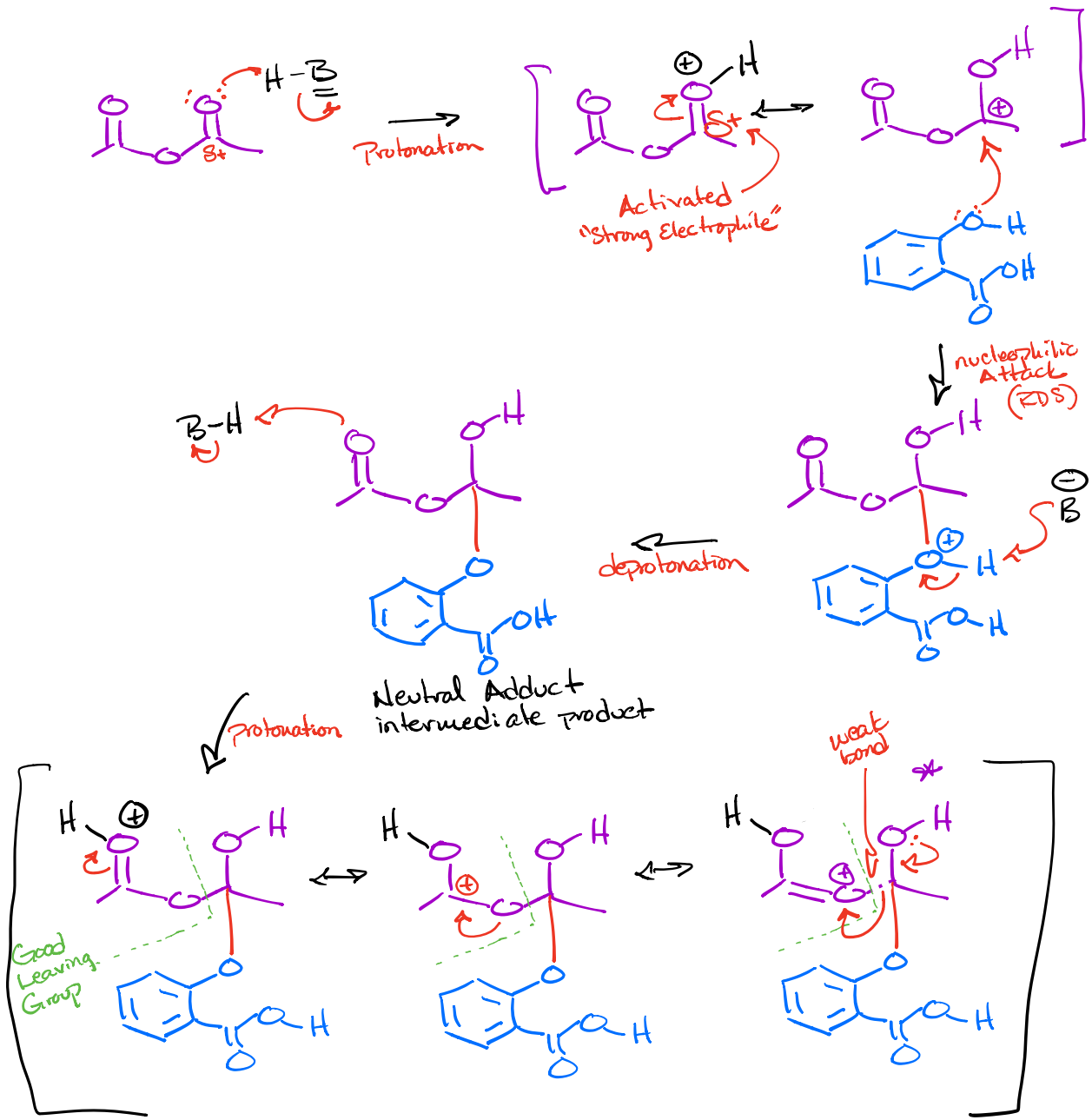
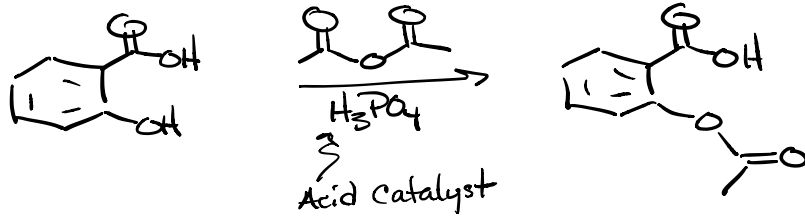


Mode of Action



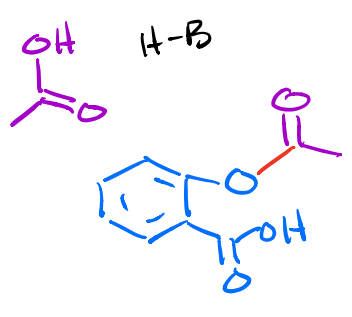
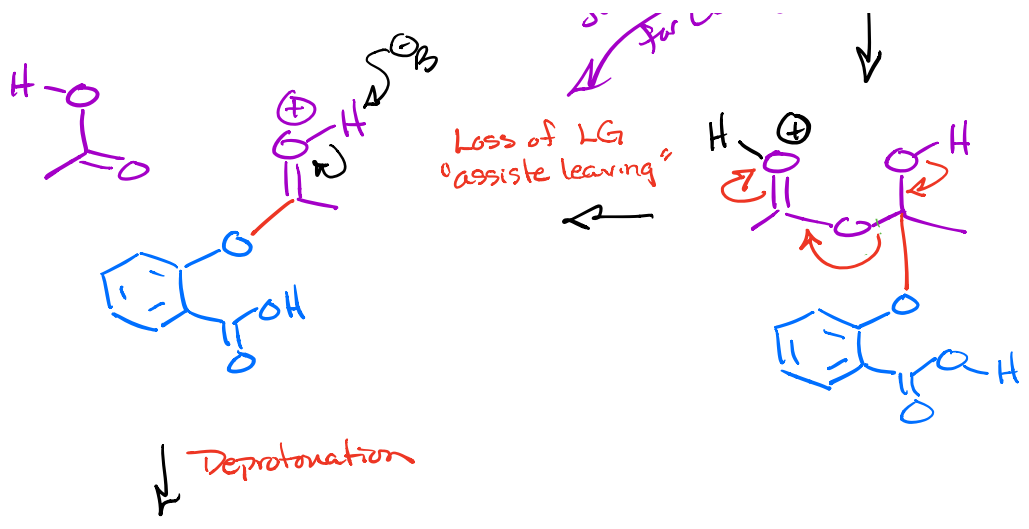


Mechanism



same step
lost repeated
clarify

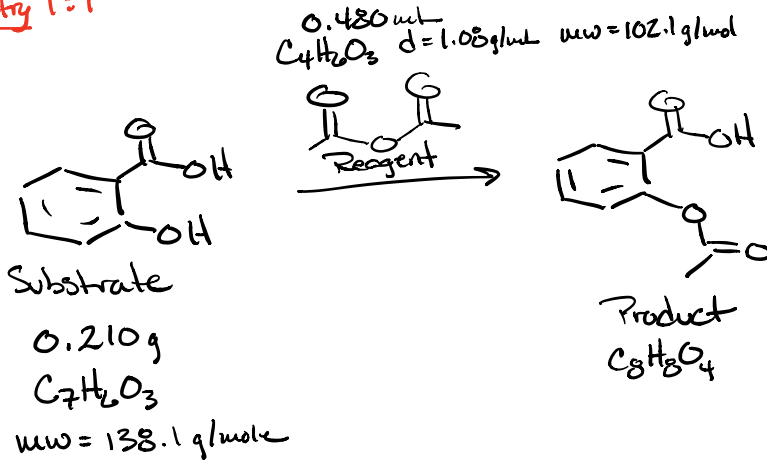
Loss of LG
"assists leaving"



Stoichiometry



Stoichiometry 1:1



what are the amounts of each reagent (mols?)

Salicylic Acid

$$0.210 \text{ g C}_7\text{H}_6\text{O}_3 \times \frac{1 \text{ mole}}{138.1 \text{ g}} \times \frac{1000 \text{ mmol}}{1 \text{ mole}} = 1.52 \text{ mmol C}_7\text{H}_6\text{O}_3$$

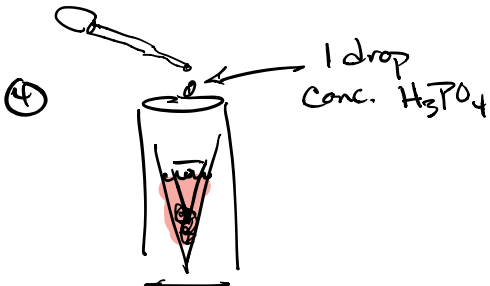
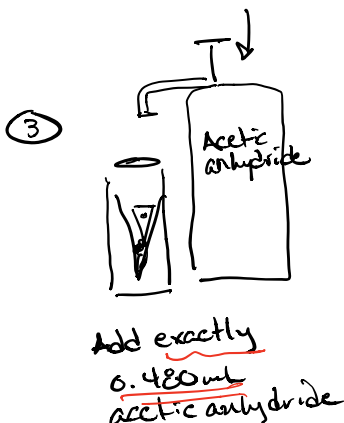
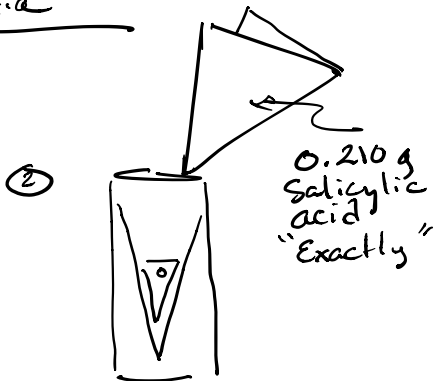
Acetic Anhydride

$$0.480 \text{ mL C}_4\text{H}_6\text{O}_3 \times \frac{1.08 \text{ g}}{1 \text{ mL}} \times \frac{1 \text{ mole}}{102.1 \text{ g}} \times \frac{1000 \text{ mmol}}{1 \text{ mole}} = \underline{\underline{5.08 \text{ mmol C}_4\text{H}_6\text{O}_3}}$$

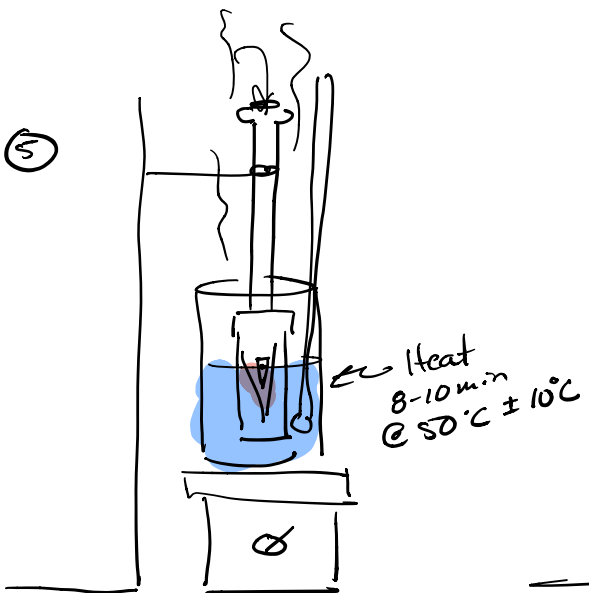
$$\frac{5.08 \text{ mmol C}_4\text{H}_6\text{O}_3}{1.52 \text{ mmol C}_7\text{H}_6\text{O}_3} = 3.34 \times \text{excess of acetic anhydride?}$$

- ① Le Chatelier's principle to increase yield
- ② $\text{C}_2\text{H}_5\text{OCOC}_2\text{H}_5 + \text{H}_2\text{O} \rightarrow 2 \text{C}_2\text{H}_5\text{OH}$ Glassware is hydrophilic
- ③ Solvent? \Rightarrow acetic anhydride is the solvent

Set up from Paria

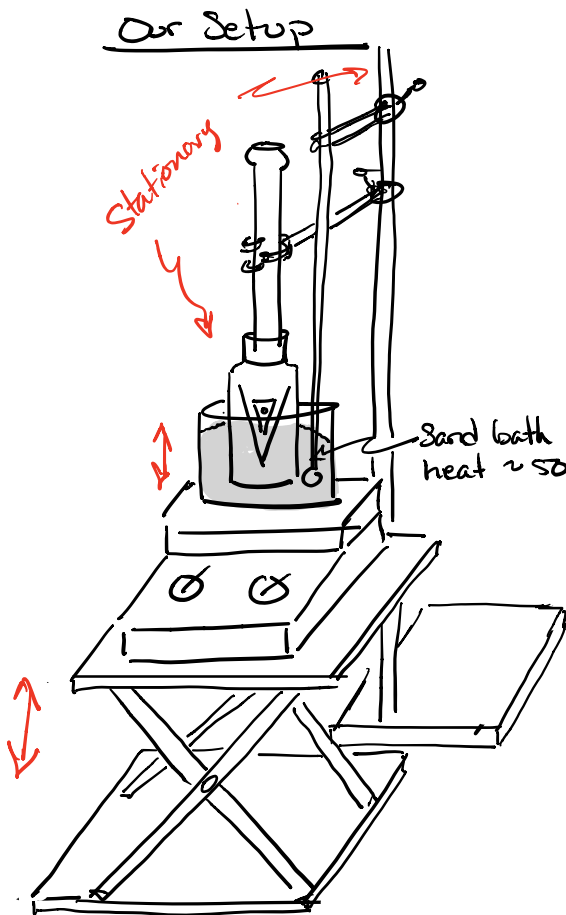


Conc HCl	12.1 M	12.1 M
Conc H_2SO_4	18.0 M	36.0 M
Conc H_3PO_4	14.8 M	44.4 M

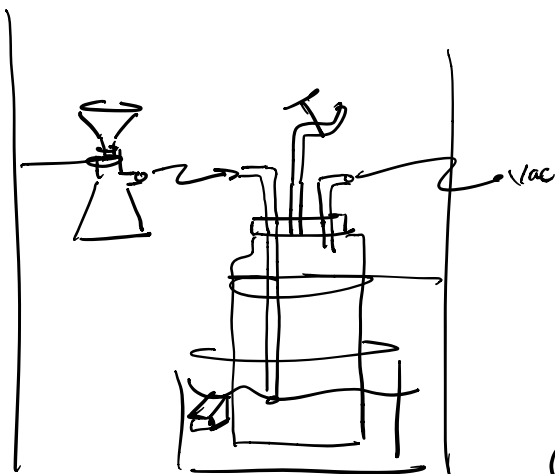


- ⑥ Cool to rt
- ⑦ Add 3 ml DI to Conical vial & stir
- ⑧ Filter on hirsch
- ⑨ Rinse w/ 1 ml 0°C DI
- ⑩ Air dry for 5-10 min
- ⑪ Tare petri dish
- ⑫ Store in petri dish
- ⑬ Take mass

Day 2
 Take mass
 Calc % yield
 Take up
 $FeCl_3$ test
 Solid FTIR



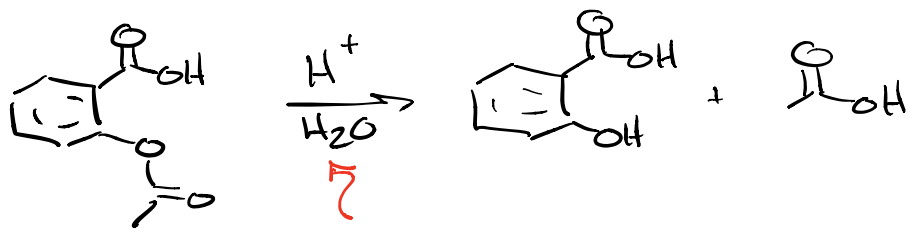
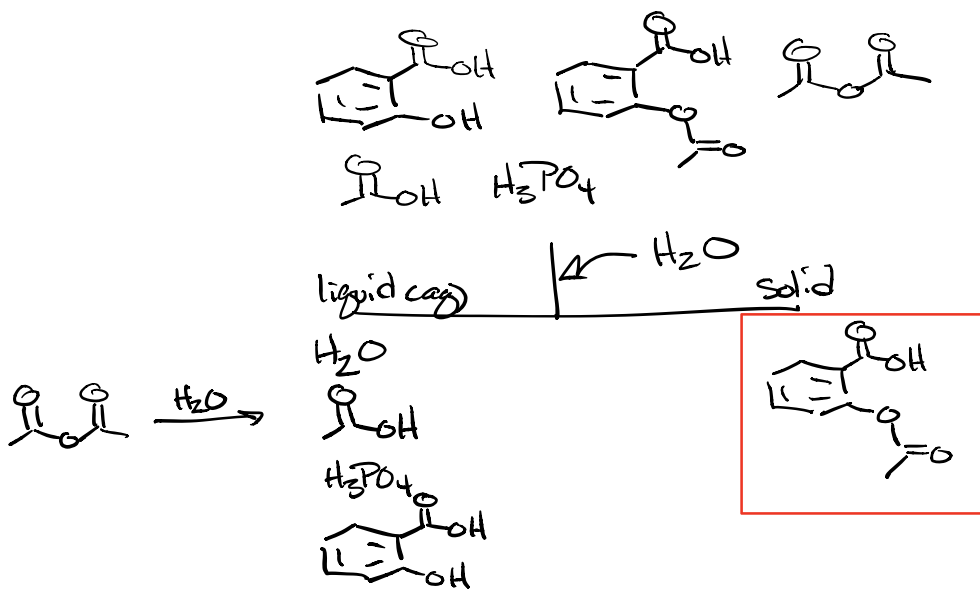
Jackstand
in elevated position
to allow for lowering



- ① Set up hot plate, Jackstand, ring stand, clamps, & sand bath.
- ② heat sand bath $\sim 50^{\circ}\text{C} \pm 10^{\circ}\text{C}$
- ③ Obtain $\sim 0.210\text{g}$ Salicylic acid
- ④ Weigh flask & Salicylic acid
- ⑤ Add $\sim 0.5\text{ml}$ acetic anhydride
 \Rightarrow neutralize pipet
- ⑥ Reweigh flask to get mass of acetic anhydride $\sim 0.5\text{g}???$
- ⑦ Add 1 drop conc. H_3PO_4
- ⑧ Heat $\sim 8-10\text{min}$
 \leftarrow start timer when salicylic acid dissolves
- ⑨ Cool to rt
- ⑩ Add $\sim 3.0\text{ml}$ H_2O
 & Stir w/ spatula
- ⑪ Filter on Hirsch
- ⑫ Rinse w $\sim 1\text{ml}$ 0°C DI
- ⑬ Air dry $5-10\text{min}$
- ⑭ Tare petri dish
- ⑮ Weigh crystals
- ⑯ Label & Store in locker

work & ...

Separation Scheme



Over rinsing
or leaving in
contact w/ H2O
too long.

After Adding DI
move quickly to
filtering \Rightarrow Stir

Make sure filtration ready to go
before adding H2O!