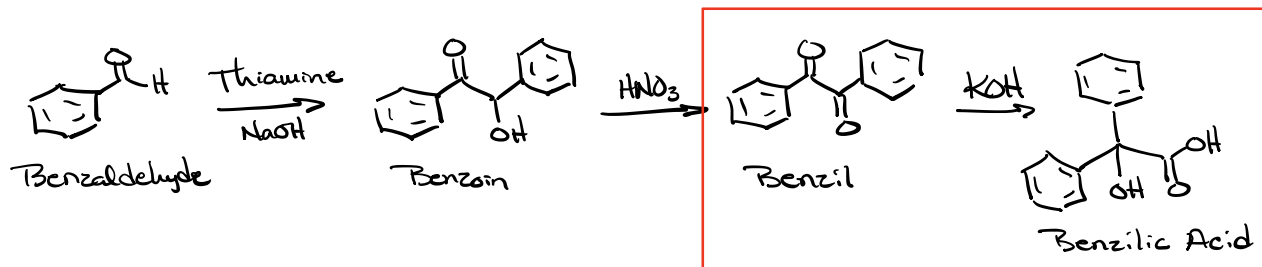
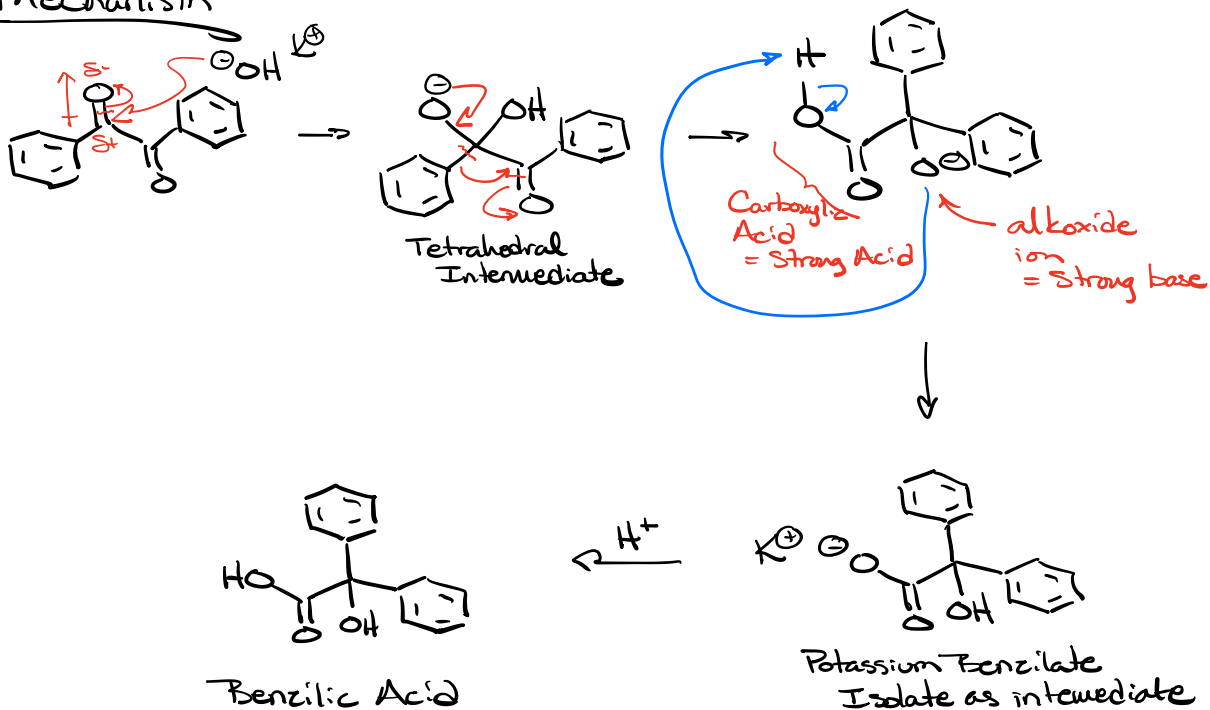


3rd Reaction of Multistep Synthesis of Benzilic Acid



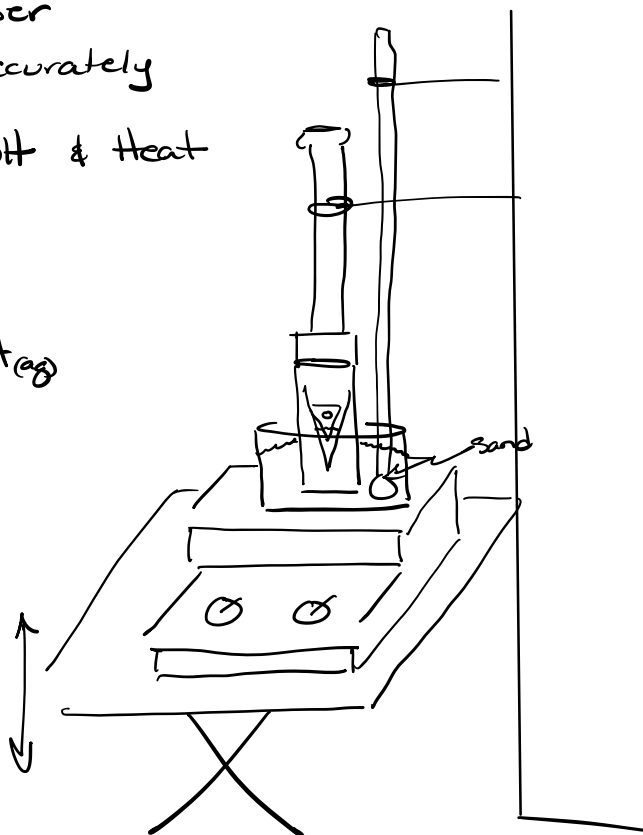
Base Catalysed Oxidation & Isomerization

Mechanism



Reaction Procedure

- Add 0.10 g benzil into 3 ml Conical vial w/ Spin vane & Air Condenser
⇒ weigh benzil accurately
- Add ~ 0.30 ml 95% EtOH & Heat ~ 100 °C until dissolved.
- Add 0.25 ml of 8 M KOH(aq) dropwise through the Condenser
- Boil Rxn ~ 100 °C - 115 °C with stirring for 15 min
- Rxn should turn a deep purple / black color
- Stop & cool Rxn to Room temp.



Work up

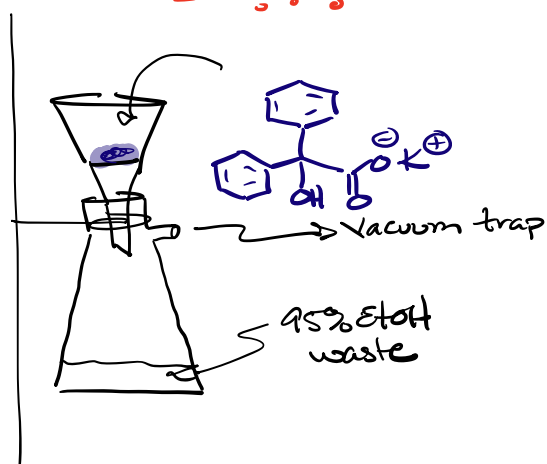
- Transfer Contents of Rxn to a 10 mL beaker



- Ice bath beaker for 15 min to induce crystallization (may need scratching Rod)

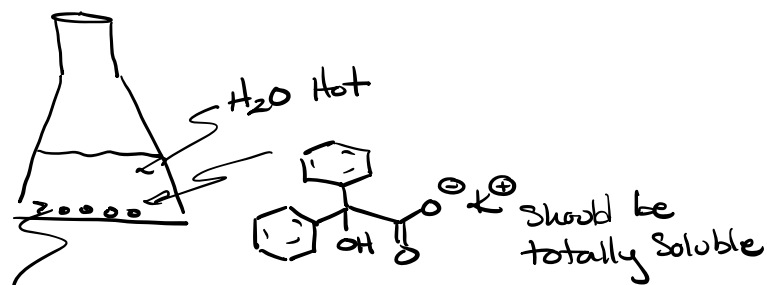
- Filter on Hirsch funnel & Rinse w/ 3 x 1 mL 95% EtOH @ 0°C

⇒ Not H₂O !!!



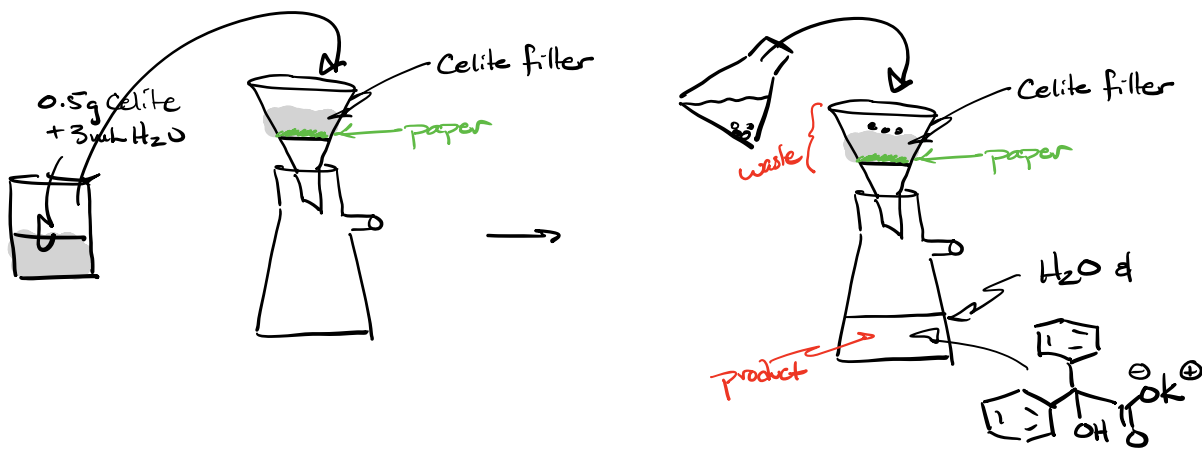
- Dry & weigh the solid
- Transfer Solid to Small 10 mL Erlenmeyer flask and add 3 mL Hot DI H₂O.
- Stir to dissolve salt

⇒ Check to see that the solution is homogeneous.

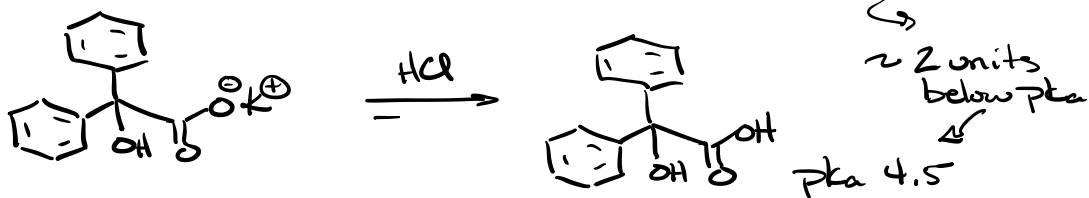


any solid is not potassium benzoate & needs to be removed before moving forward.

⇒ if solids present, filter out solids using a hirsch funnel w/ celite filter.



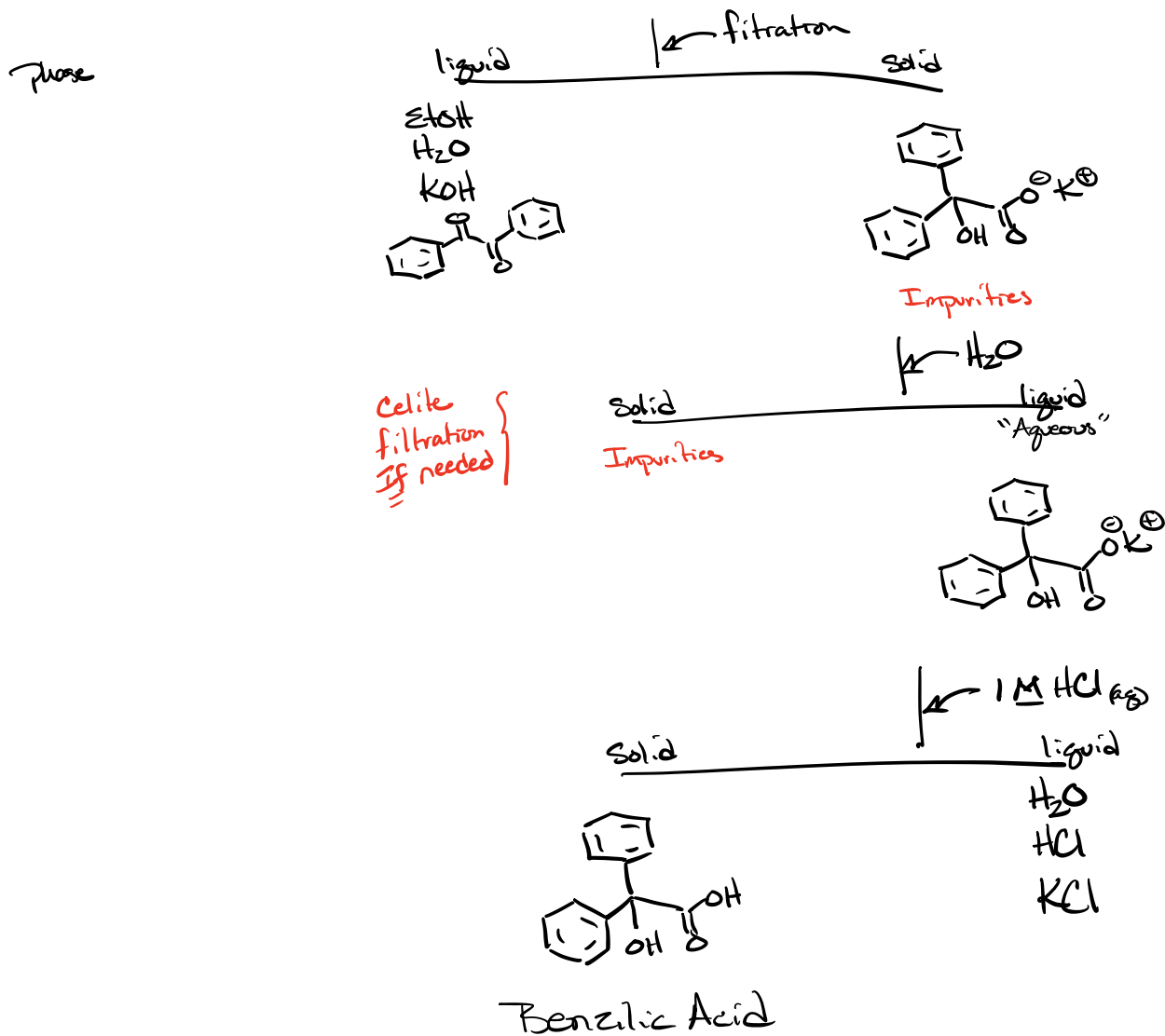
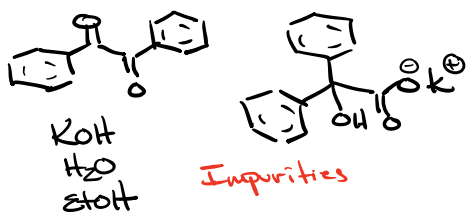
- Acidify solution with 0.5 mL of 1M HCl(aq). Take pH down to 2.



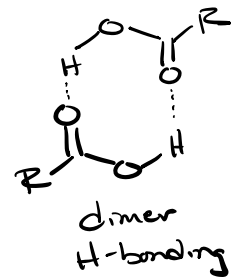
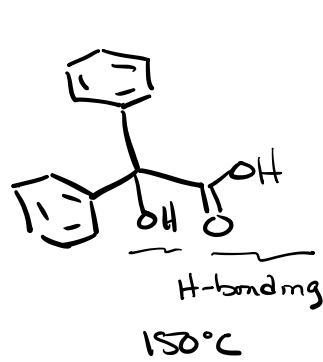
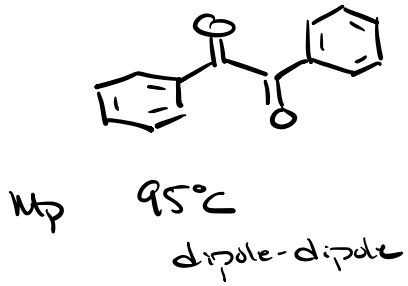
- Ice 15 min
 - Filter on Hirsch funnel, Rinse w/ 2 x 1 mL H₂O
 - dry
 - mass
 - melting point
 - FTIR
- } Characterization

Separation Scheme

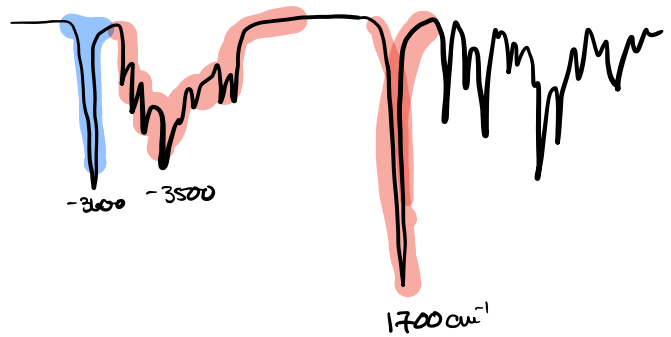
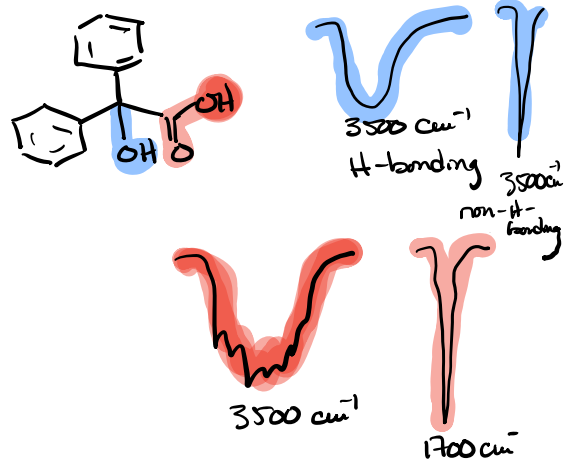
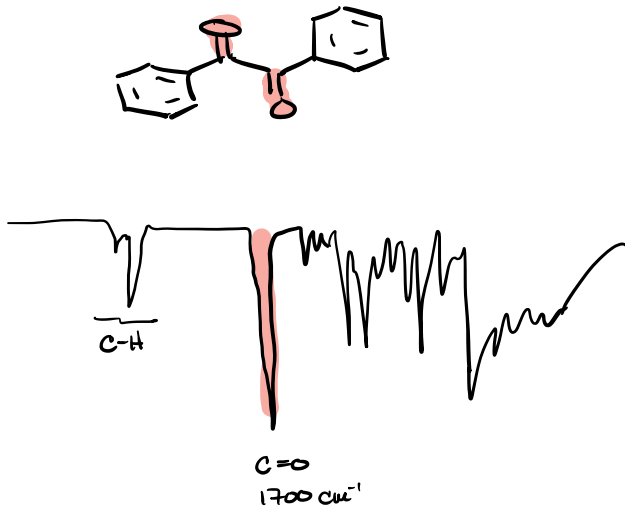
Starting materials
products
possible side-products



Melting Point



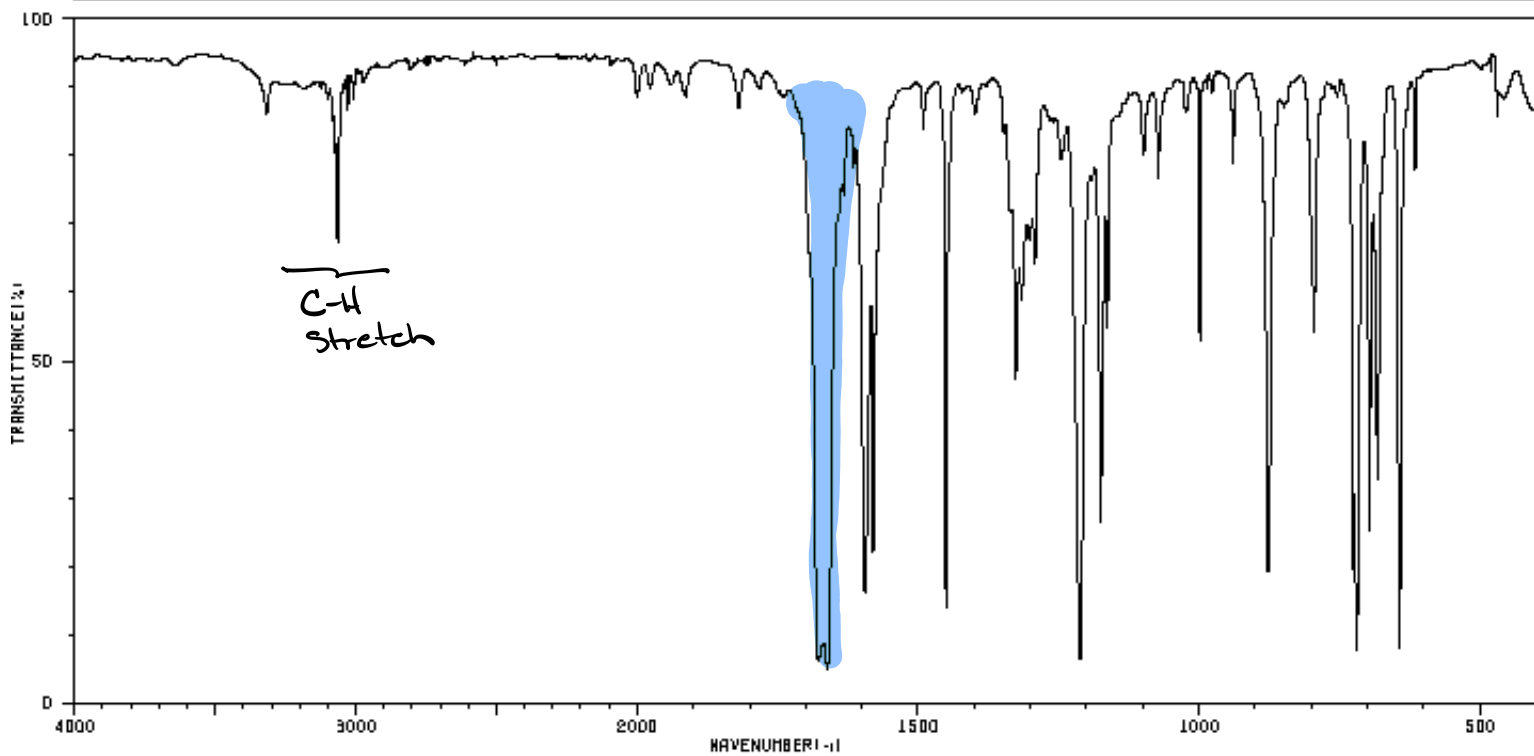
FTIR



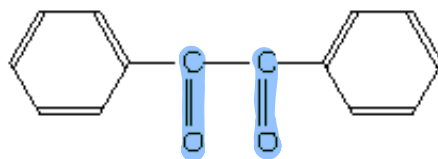
HIT-NO=1146 SCORE= () SDBS-NO=871 IR-NIDA-47574 : KBR DISC

BENZIL

C₁₄H₁₀O₂

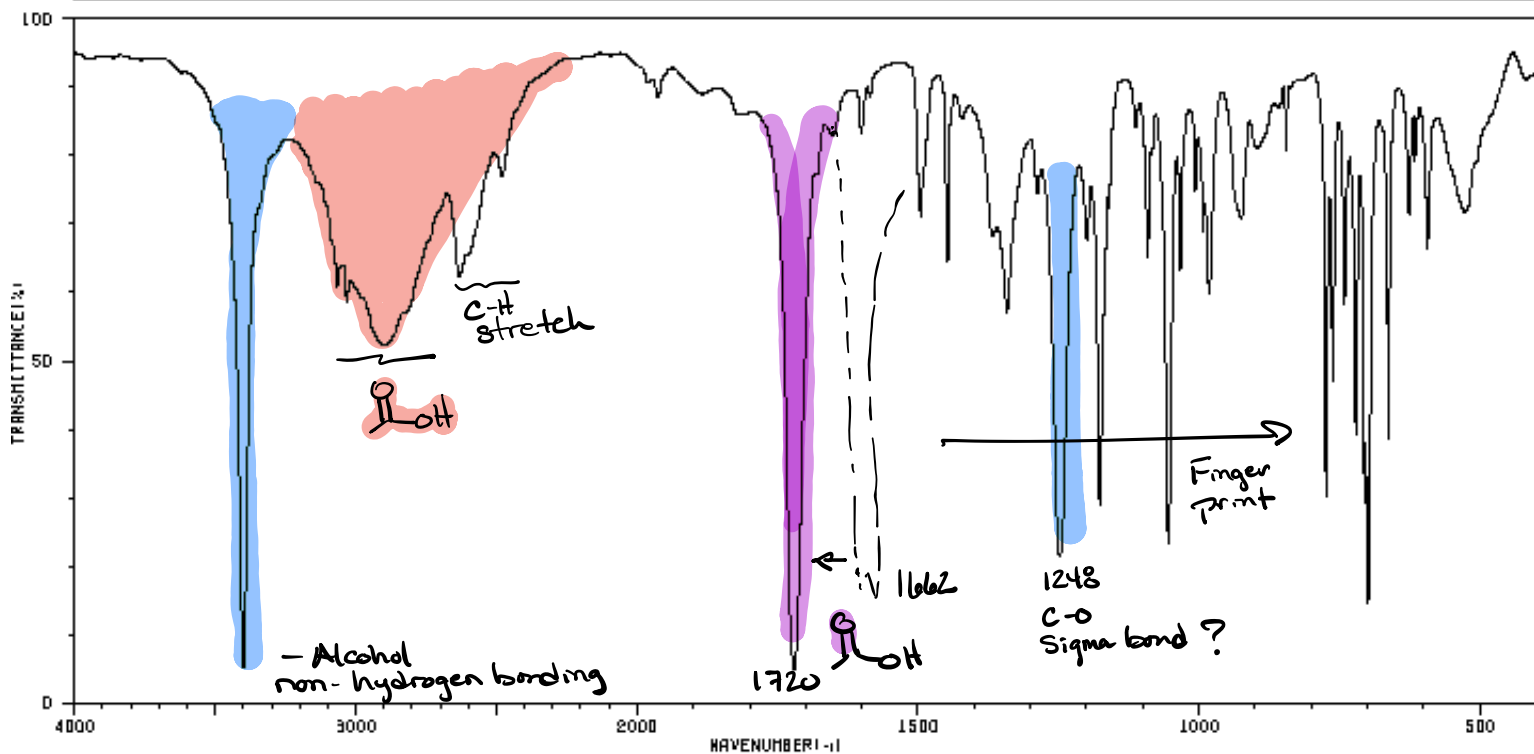


3076	77	1633	72	1326	46	1163	62	725	18
3065	84	1615	74	1316	57	1098	77	720	7
2001	84	1595	15	1303	84	1073	74	697	23
1914	84	1580	21	1292	62	999	60	682	31
1677	5	1491	81	1246	77	940	77	644	7
1668	8	1451	19	1212	6	878	18	615	74
1662	4	1348	78	1176	25	796	62	469	81



BENZILIC ACID

C₁₄H₁₂O₃



3400	6	1720	4	1200	64	993	66	721	37
3066	58	1495	68	1177	27	982	57	706	28
3033	57	1447	62	1160	70	925	68	700	13
2900	60	1369	66	1091	62	846	77	663	37
2630	60	1343	55	1055	22	774	29	627	68
2604	62	1289	72	1033	60	762	44	594	64
2479	74	1248	20	1007	72	742	66	527	68

