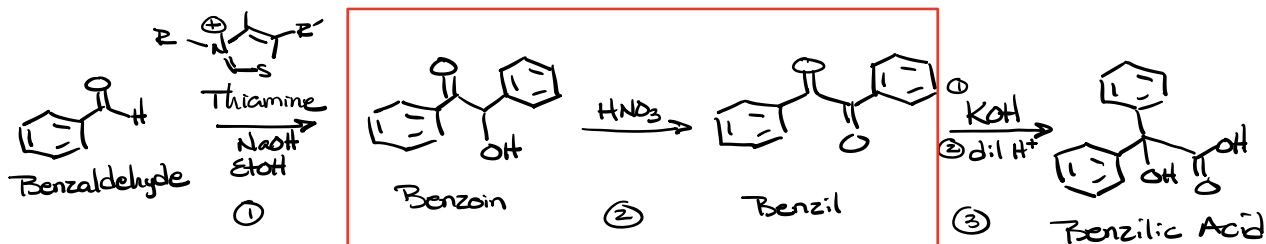
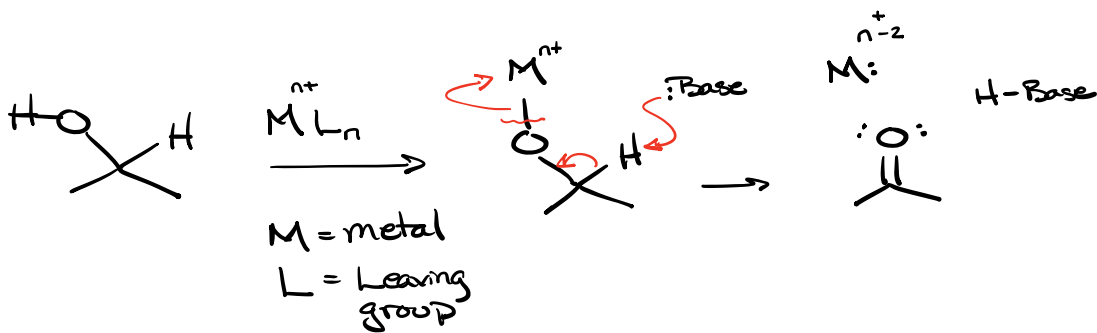


# Multistep Synthesis of Benzilic Acid

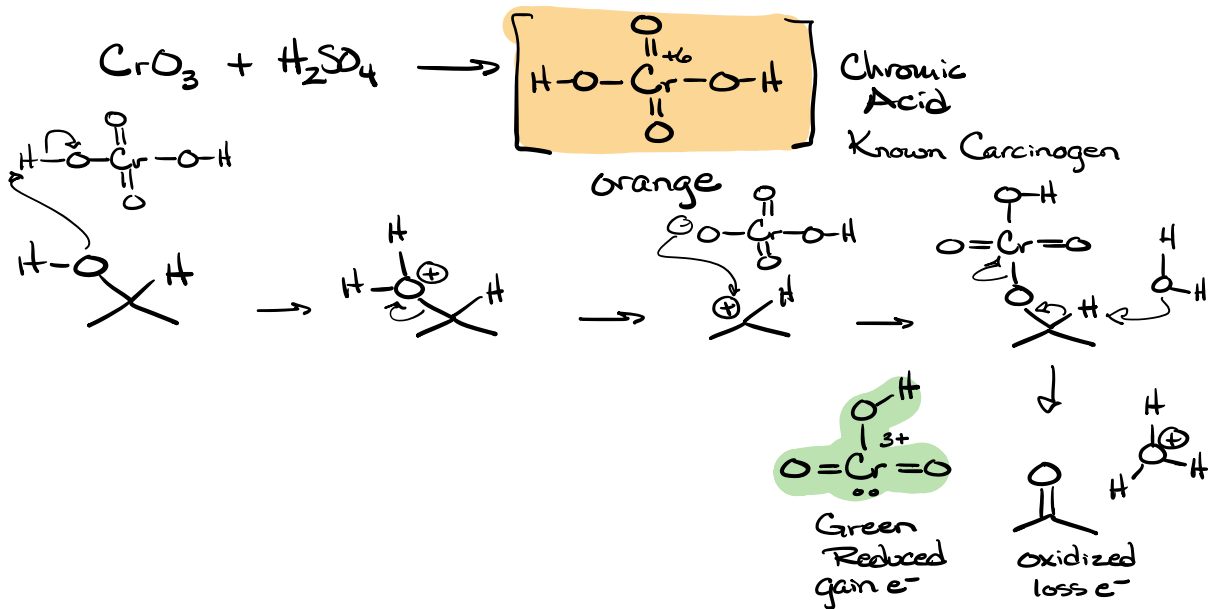


## Oxidation



## Strong Oxidizing Agents

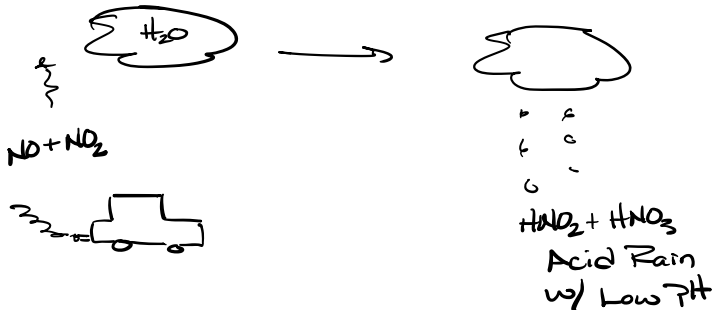
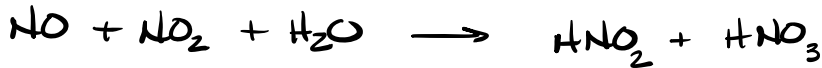
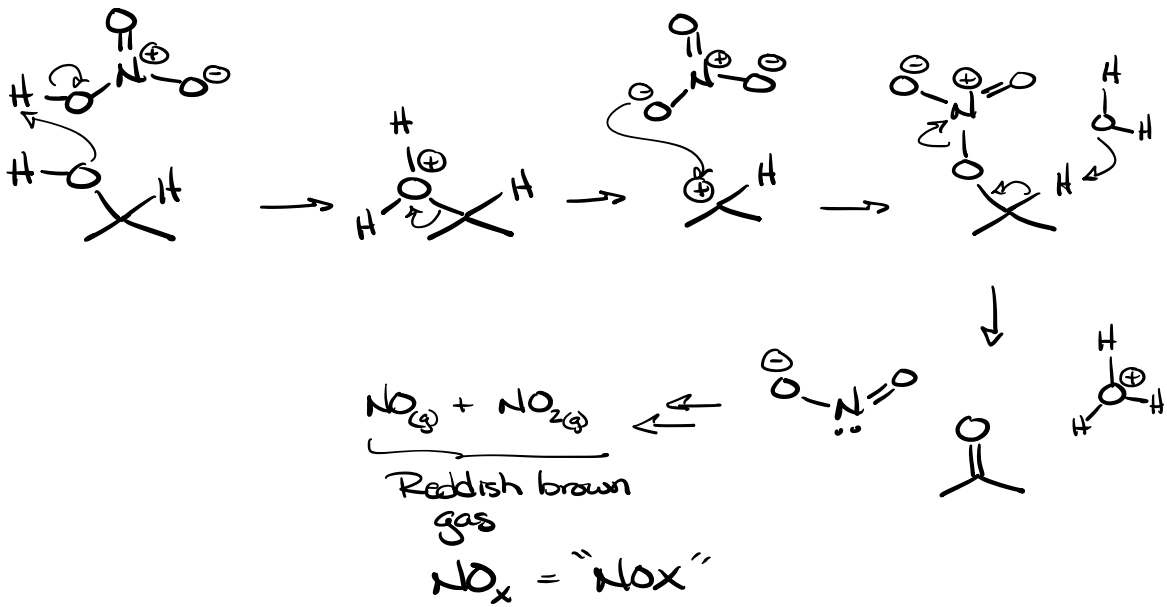
Chromic Acid - Jones Reagent "Jones"

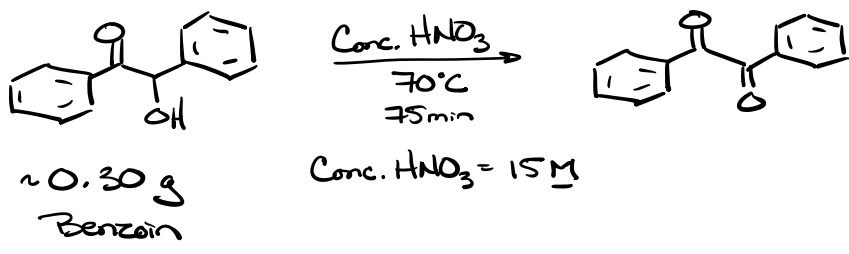


$\text{Cr}^{6+}$  hexavalent Chromium Carcinogenic

$\text{Cr}^{3+}$  Chromic Ion Chromium(III) } not  
 $\text{Cr}^{2+}$  Chromous Ion Chromium(II) } Carcinogenic

### Oxidation using $\text{HNO}_3$



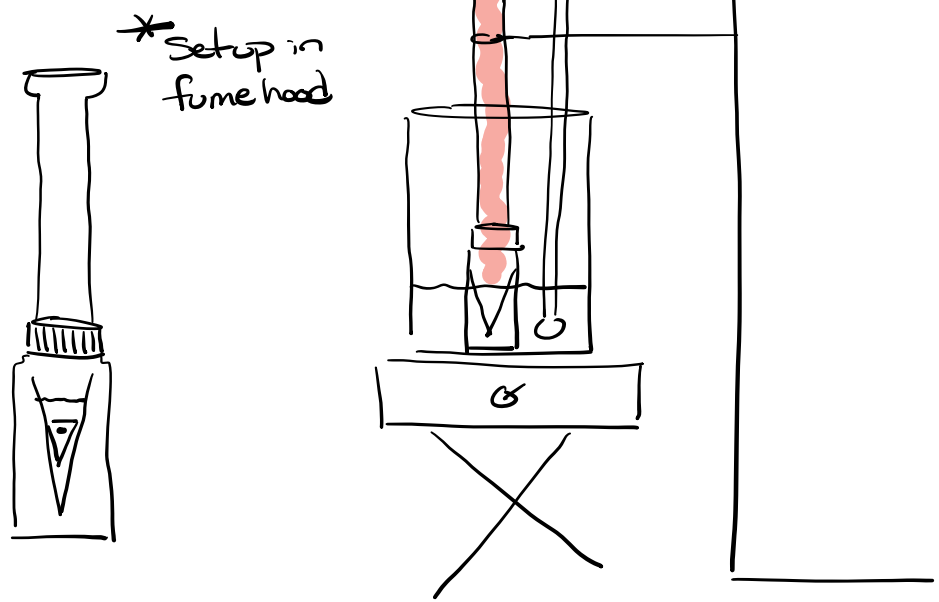


Reaction

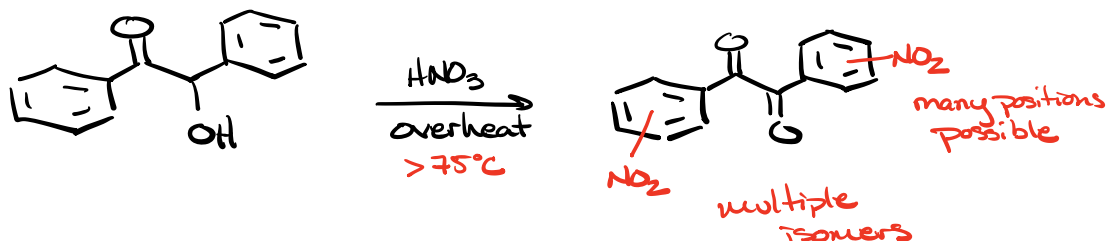
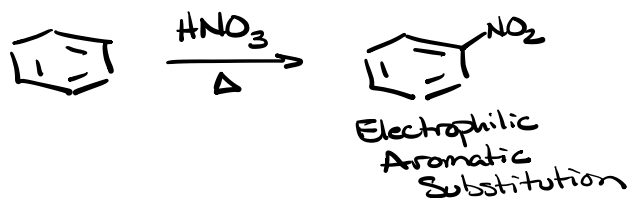
- Weigh out  $\sim 0.30 \text{ g}$  Benzoin & place into 5ml Conical Vial



- Add Spin Vane & Air Condenser
- Set up a  $\text{H}_2\text{O}$  bath & heat to  $70^\circ\text{C} \pm 5^\circ\text{C}$
- Add 1.5ml Conc.  $\text{HNO}_3$  to the Conical Vial
- Heat for 75min

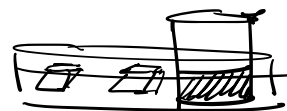
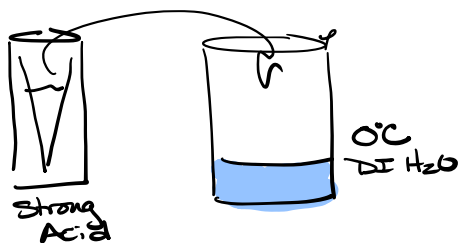


## Possible Side products

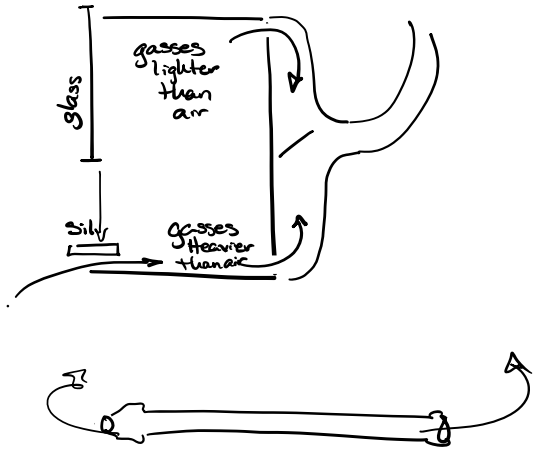
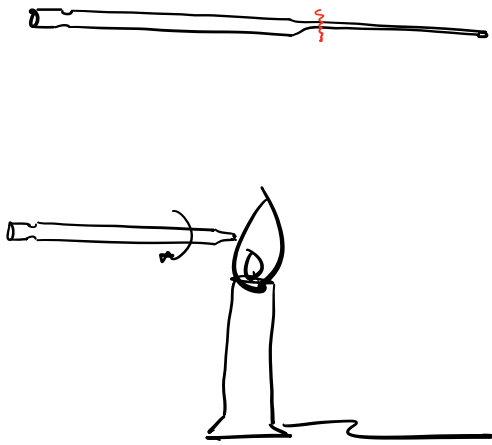


## workup

- ① - Cool Rxn to room temp
- ② - Disconnect the air condenser in fume hood (& leave the condenser in hood to air out)
- ③ - Transfer the Reaction to 25ml-Beaker w/  
4 ml 0°C DI H<sub>2</sub>O.  
Cold



- ④ - Rinse Conical vial & Spin vane w/ DI H<sub>2</sub>O & Add to 25-ml Beaker
- ⑤ - Ice bath beaker for 10-15 min to induce Crystallization  
\* (Scratch glass if needed)



Silicate glass  
Scratching Rod  
"Soft"

### Two types of glass

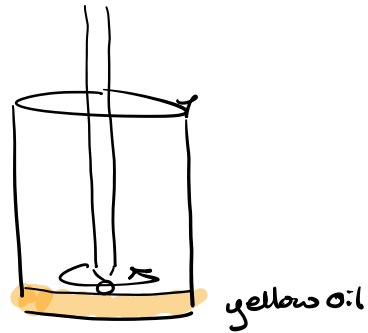
Borosilicate  
"Pyrex"  
Low Coefficient of expansion  
"Hard"

Silicate Normal  
High Coefficient of expansion  
"Soft"



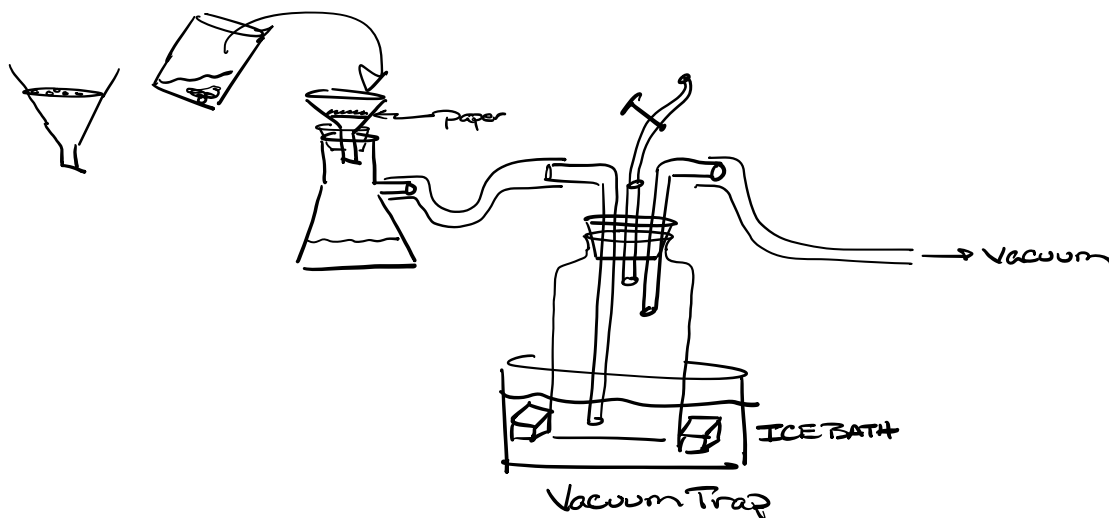
Glass shards  
Nucleation sites for crystallization  
"Seed Crystals"

Borosilicate



workup Cont.

- ⑥ - Filter on Hirsch funnel & Rinse with ~5ml 0°C DI H<sub>2</sub>O

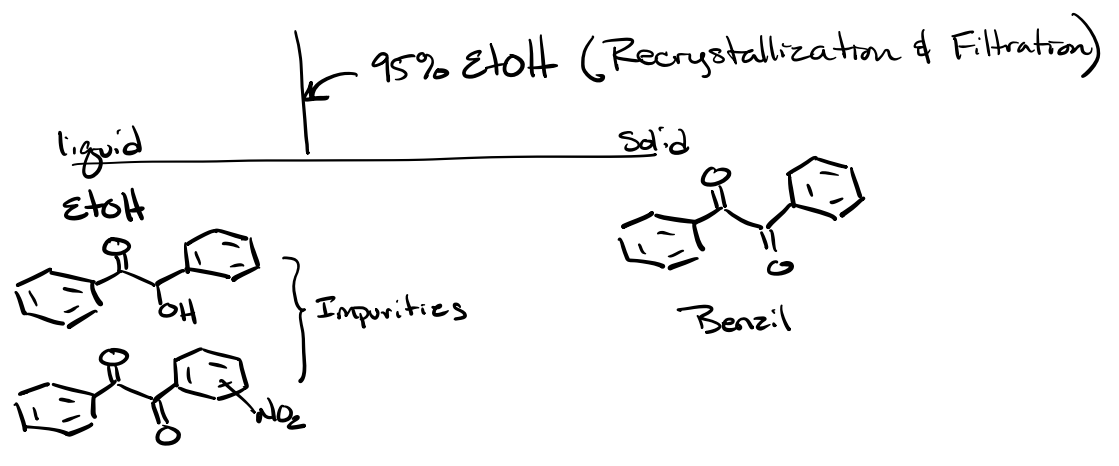
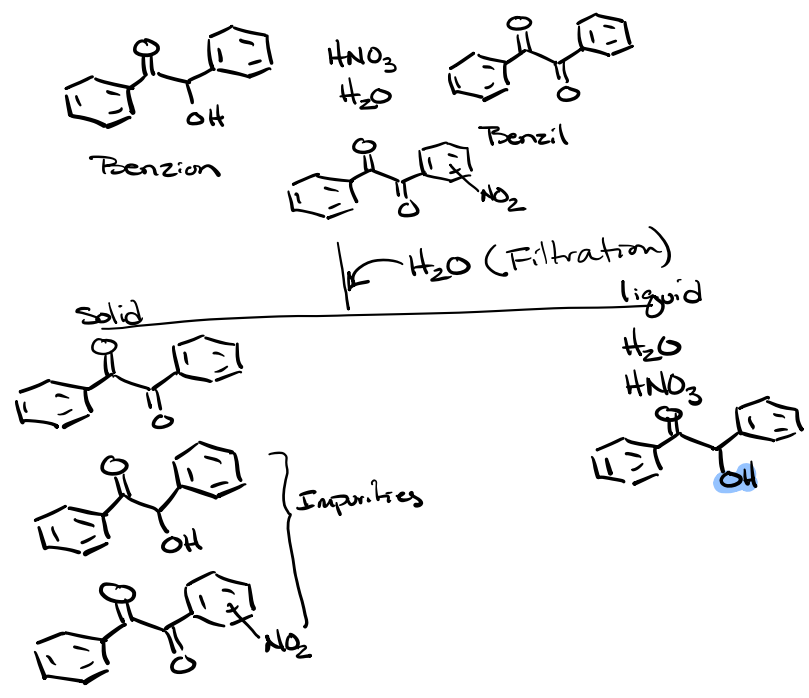


⑦ weigh the solid obtained

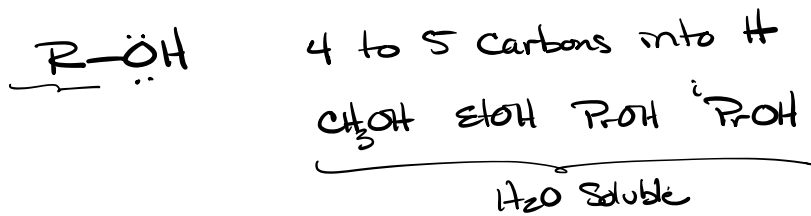
⑧ Recrystallize from 95% EtOH

- Characterization
- ⑨ weigh final solid → Calc % isolated yield
  - ⑩ melting point
  - ⑪ FTIR Spectroscopy

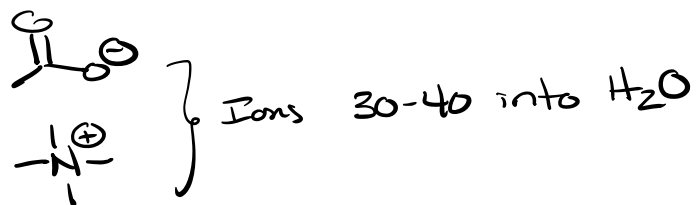
# Separation Scheme



Different functional groups allow for the solubility of Carbons into H<sub>2</sub>O

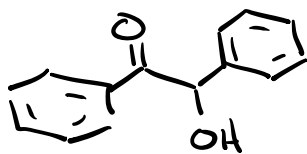


BuOH  
Limited  
H<sub>2</sub>O Solubility

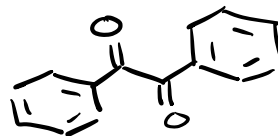




## Melting Point

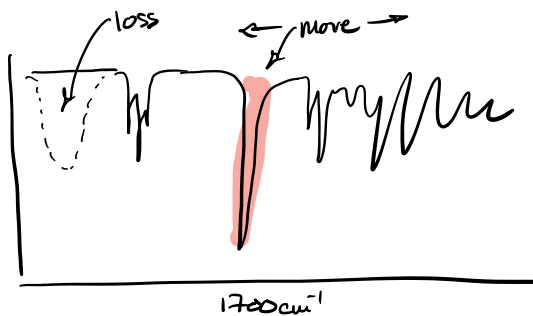
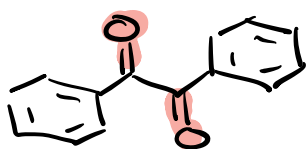
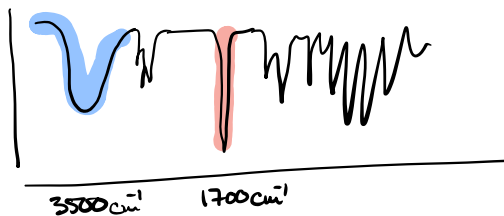
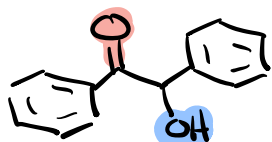


Benzoin  
MP 137°C  
H-bonding



Benzil  
Lower 94.8°C  
dipole-dipole

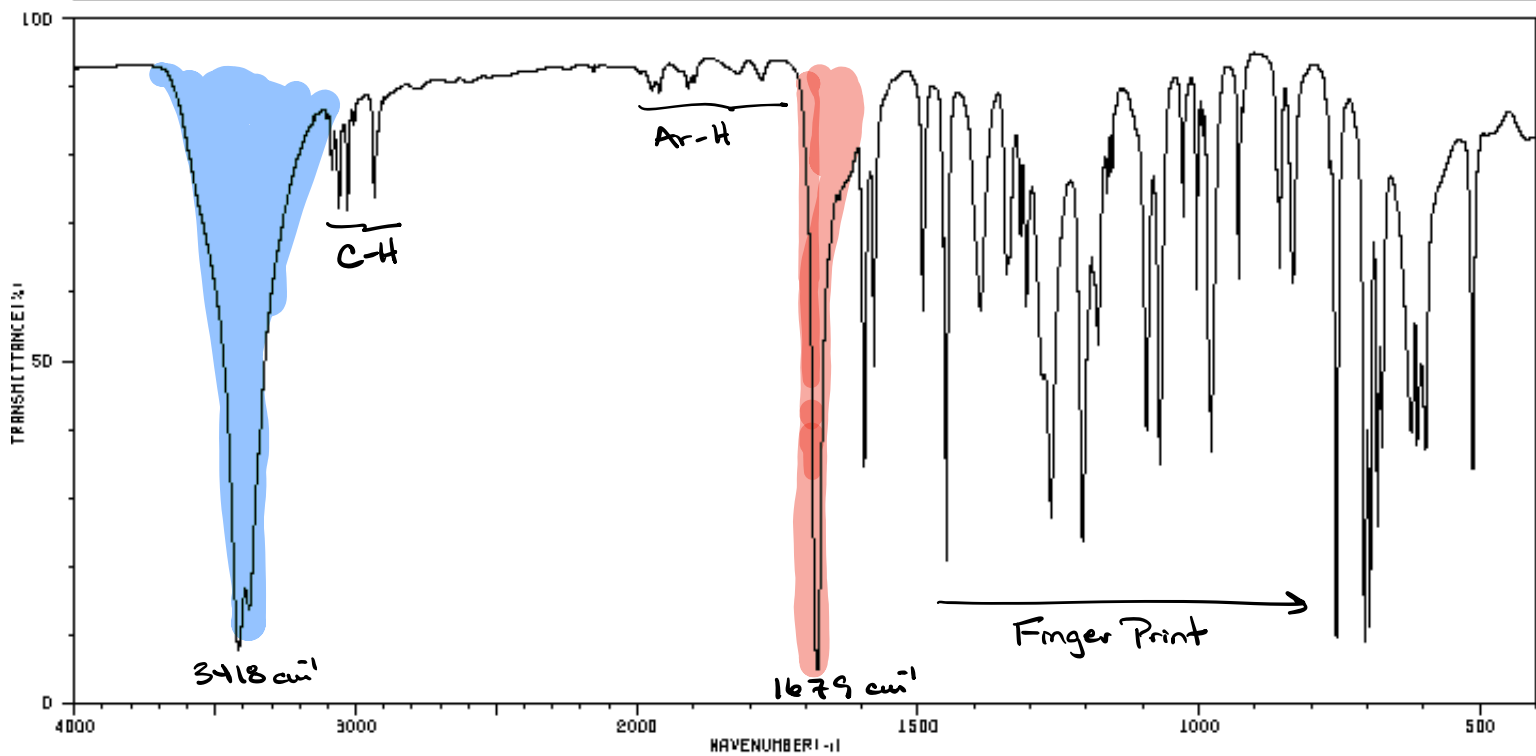
## IR Spectroscopy



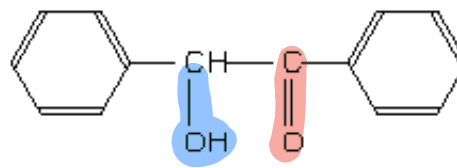
HIT-NO=1634 SCORE= ( ) SDBS-NO=1700 IR-NIDA-62015 : KBR DISC

BENZOIN

C<sub>14</sub>H<sub>12</sub>O<sub>2</sub>

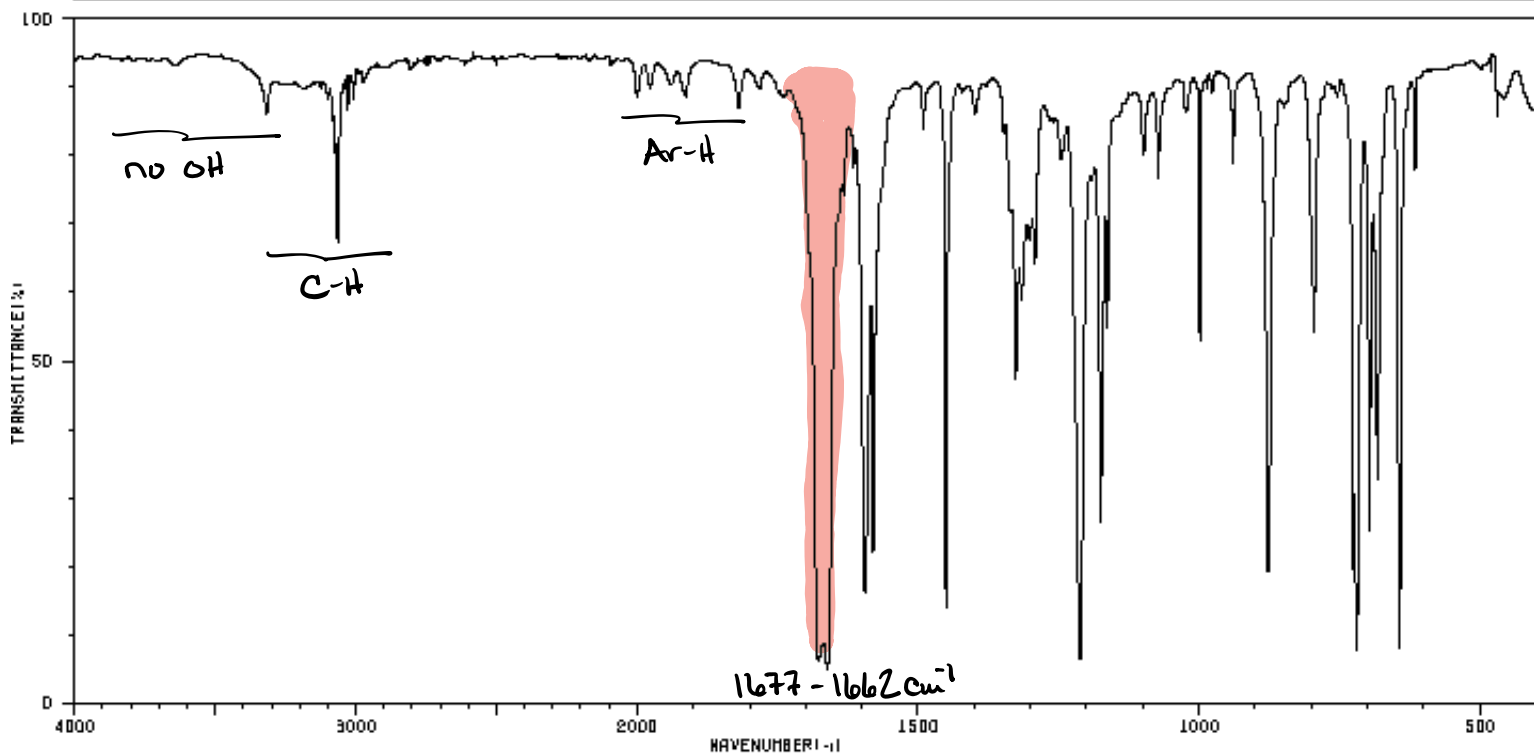


3418	7	1456	64	1263	26	983	47	696	10
3380	13	1450	20	1208	23	978	35	683	25
3061	70	1390	55	1180	50	929	60	675	36
1679	4	1343	60	1093	38	866	62	622	38
1596	33	1318	66	1070	34	833	58	613	36
1579	47	1308	55	1028	88	756	9	597	35
1492	65	1278	46	1006	68	706	8	513	33



BENZIL

C<sub>14</sub>H<sub>10</sub>O<sub>2</sub>



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	26	796	62	469	81

