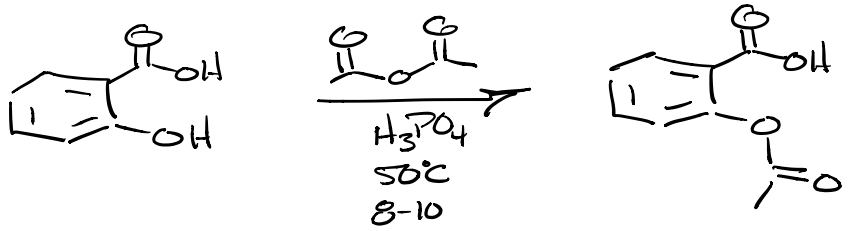
Product Characterization

Purity vs. Characterization

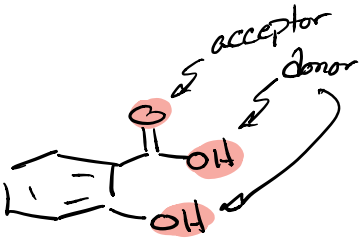
100%
one material
Chromatography
Side products
Amounts

MP
Chemical test
Spectroscopy — NMR ^{13}C, ^{1}H; FTIR; Mass Spec
Chromatography — GC-mass spec



MP
$FeCl_3$
FTIR
- How to sample a solid
- Spectrum analysis

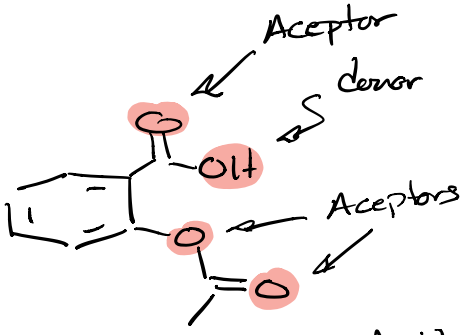
Melting Point



Salicylic Acid

Hydrogen bonding

157 - 158 °C



Acetylsalicylic Acid

Less H-bonding ↓

more mass ↑

C_2H_2O 48 g/mol

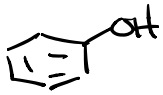
134.5 - 135.5 °C

134.5 - 135.5 °C ⟹ product is as expected

128 - 132 °C ⟹ Broad & depressed from Lit value
⟹ impure & Contains Starting material

147 - 154 °C ⟹ Broad & depressed from Starting material
⟹ Majority of recovered product is starting material
acetylsalicylic acid impurity in starting material
⟹ Run failed

Ferric Chloride test

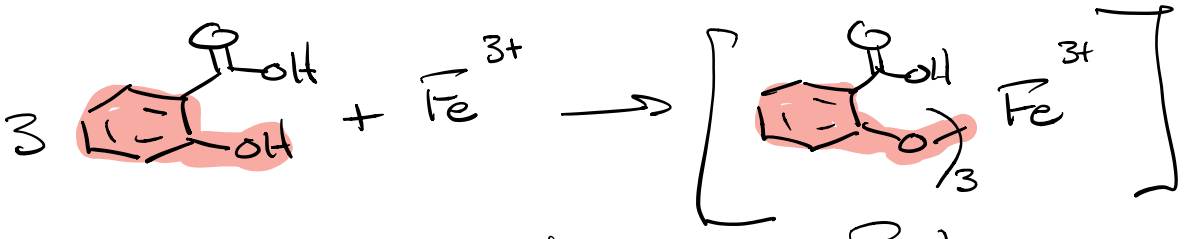
Test for phenol 

Ferric ion Fe^{3+} (Iron(III))

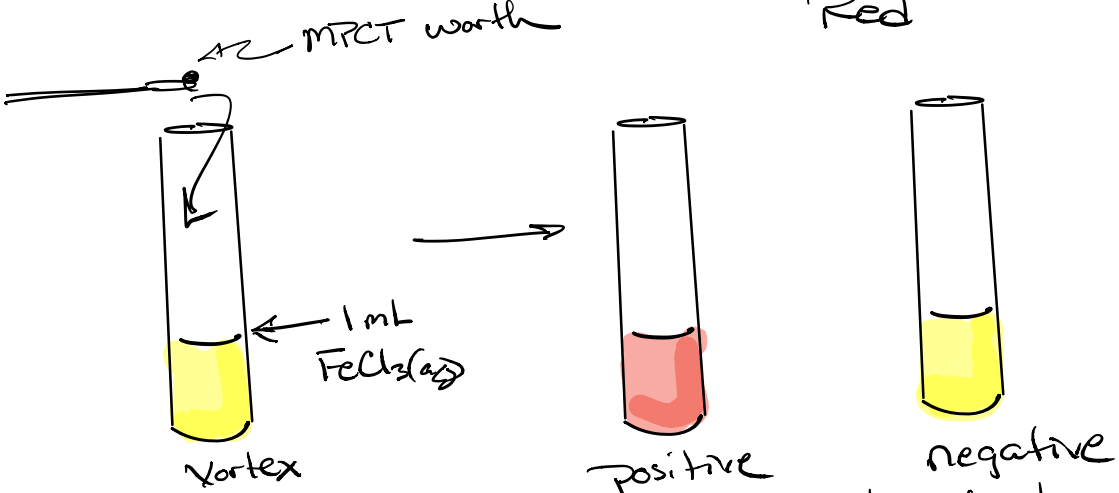
Ferrous ion Fe^{2+} (Iron(II))

$FeCl_{3(aq)}$ Clear & yellow tinge

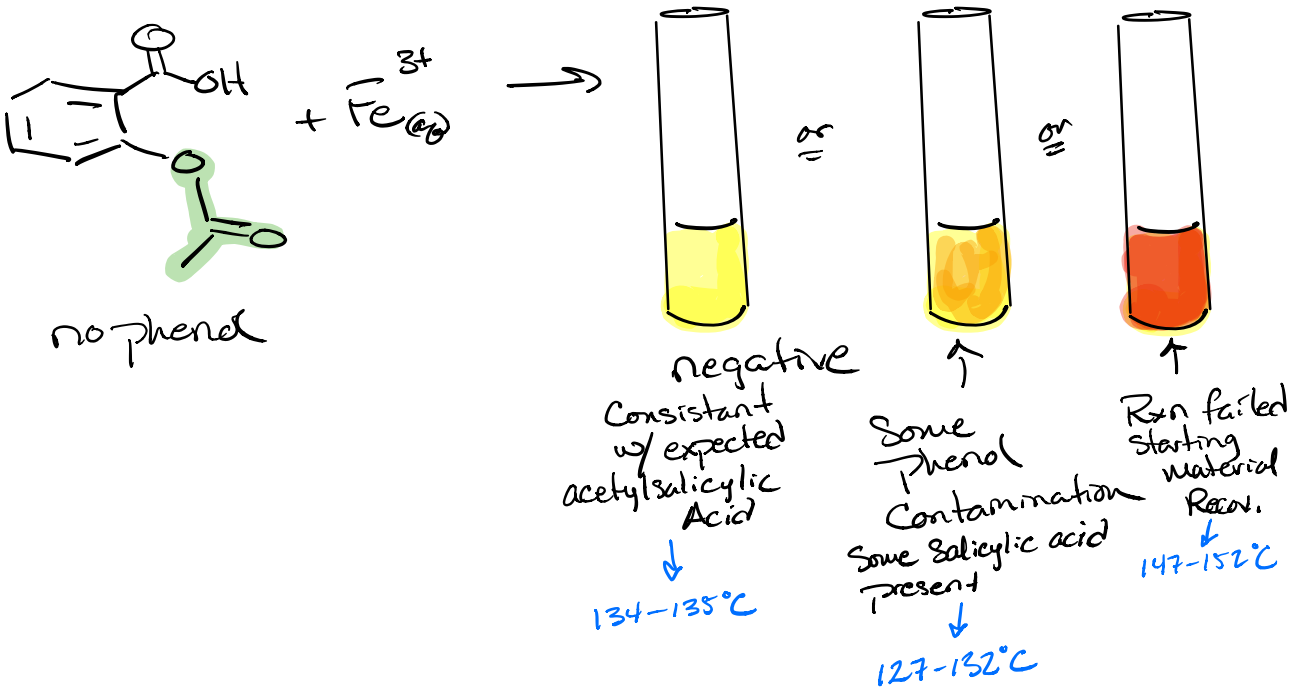
Deep Red Color
magenta

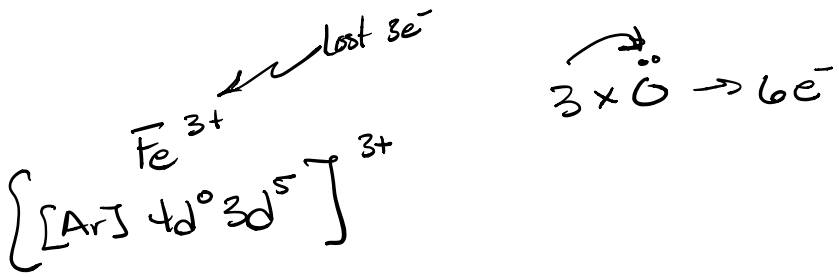


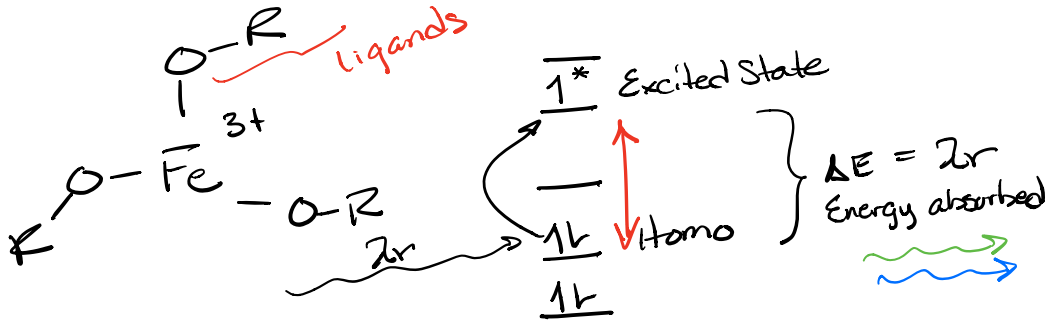
Red

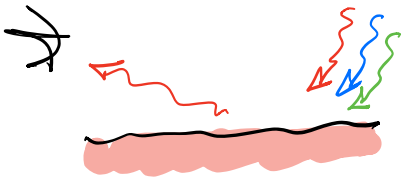


Colormetric test

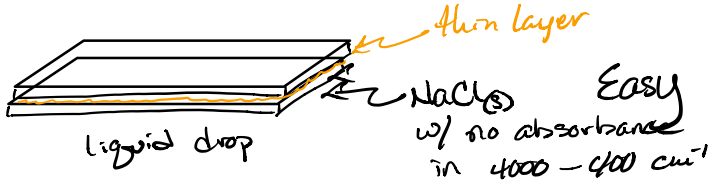

OH
+ Fe 3+
no phenol
negative
Consistant w/ expected acetylsalicylic Acid
134–135°C
or
Some Phenol Contamination
Some Salicylic acid present
127–132°C
or
Rxn failed Starting material Recov.
147–152°C


Lost 3e⁻
Fe 3+
[[Ar] 4d⁰ 3d⁵] 3+
3 x O → 6e⁻


O–R
Ligands
3+
O– Fe –O–R
R
2r
1*
Excited State
1r
1r
Homo
ΔE = 2r
Energy absorbed



Solid phase FTIR

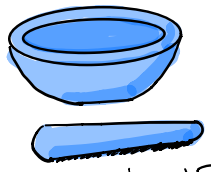


Easy
w/ no absorbance
in 4000 – 400 cm^{-1}

Solid KBr Pellet

KBr plate

1. Cheap
2. Anhydrous
3. No IR absorbance 4000 – 650 cm^{-1}
4. Makes good pellets

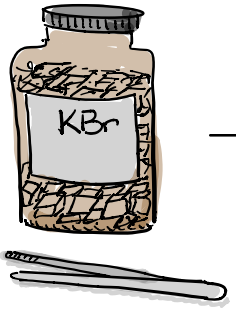


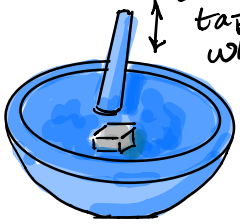
Agat Mortar & Pestle

1cm

KBr Cuttings

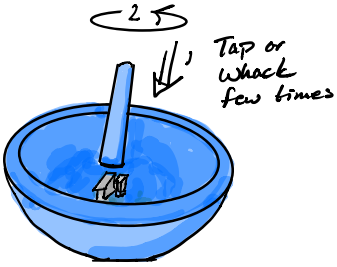
Less surface area than powder





Sharp tap whack it

Select ⅓ of crystal put rest back

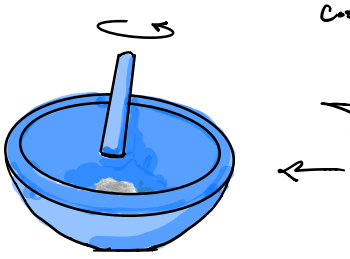


2

Tap or whack few times

Coarser salt → table salt → fine salt → talc powder

Grinding ice in 3urs

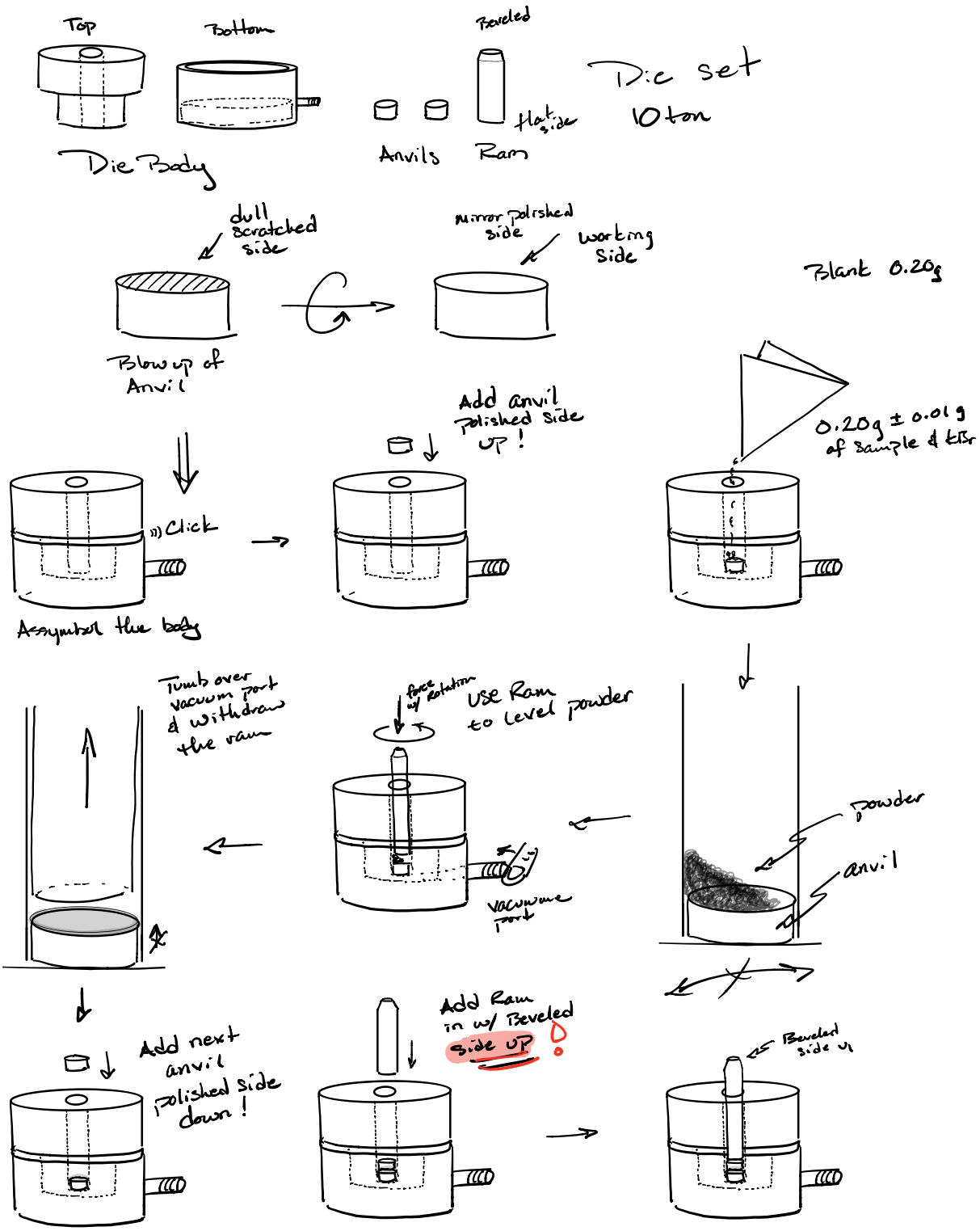


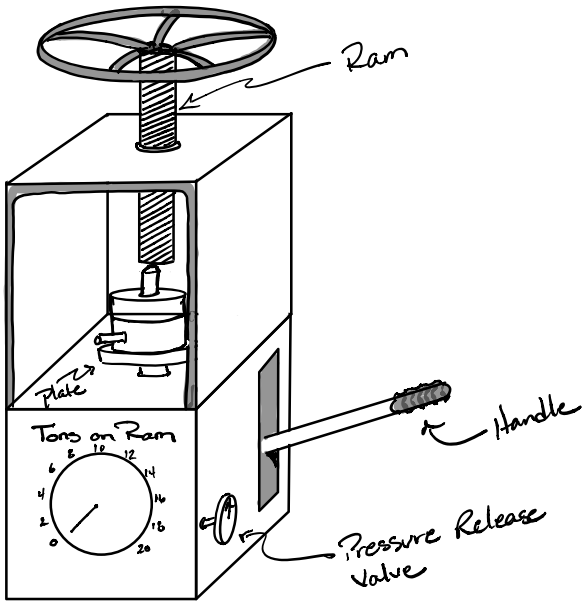
Ideal size powder dull no glinting

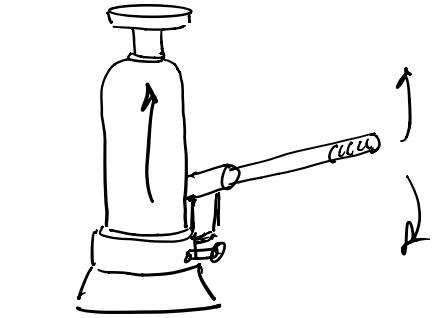
MPCT worth

Regrind to distribute sample homogeneously

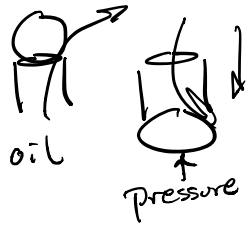
1% by mass of sample


Top
Bottom
Beveled
Die Body
Anvils
Ram
flat side
Die set
10 ton
dull scratched side
Mirror Polished side
working Side
Blank 0.20g
Blow up of Anvil
Add anvil polished side up!
0.20g ± 0.01g of Sample & KBr
Click
Assymbel the body
Tumb over vacuum port & withdraw the ram
Use Ram to Level powder
force w/ rotation
vacuume port
powder
anvil
Add next anvil polished side down!
Add Ram in w/ Beveled side up
Beveled side up

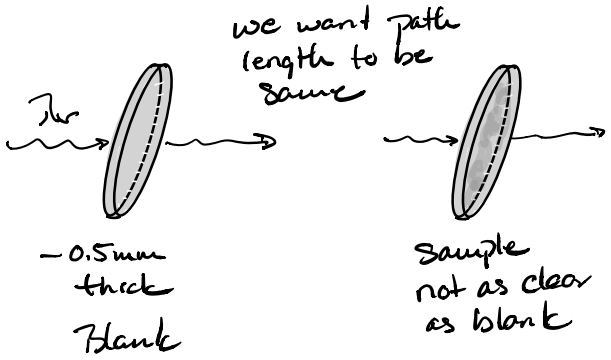




Hydralic House Jack



1. Close pressure valve clockwise
2. Insert & center the die
3. Bring ram down to contact with die ram
4. Crank up to 10 tons
   1-2 min
5. Release pressure
   1/4 turn counter clockwise of valve

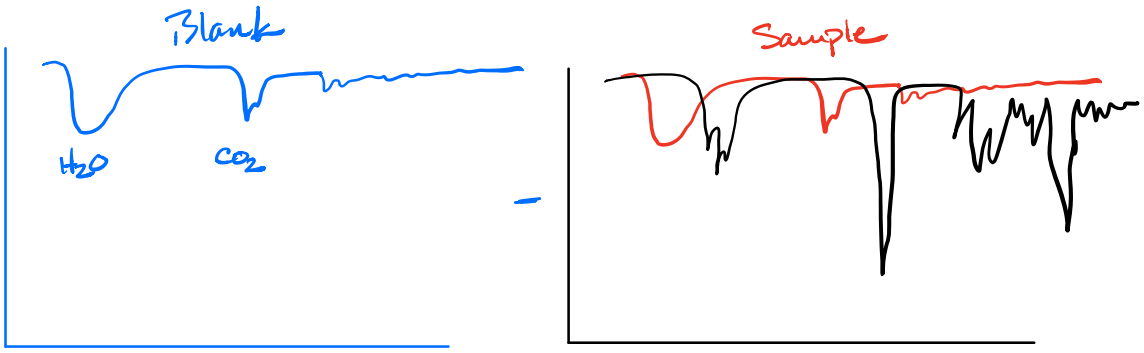


$H_2O_{(g)}$ & $CO_{2(g)}$

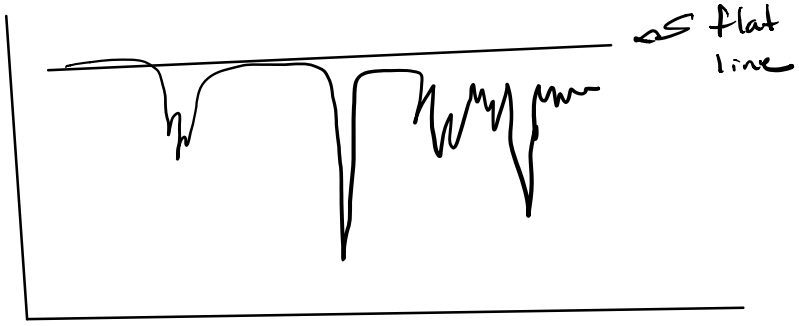
3400-3200 cm^{-1} Strong

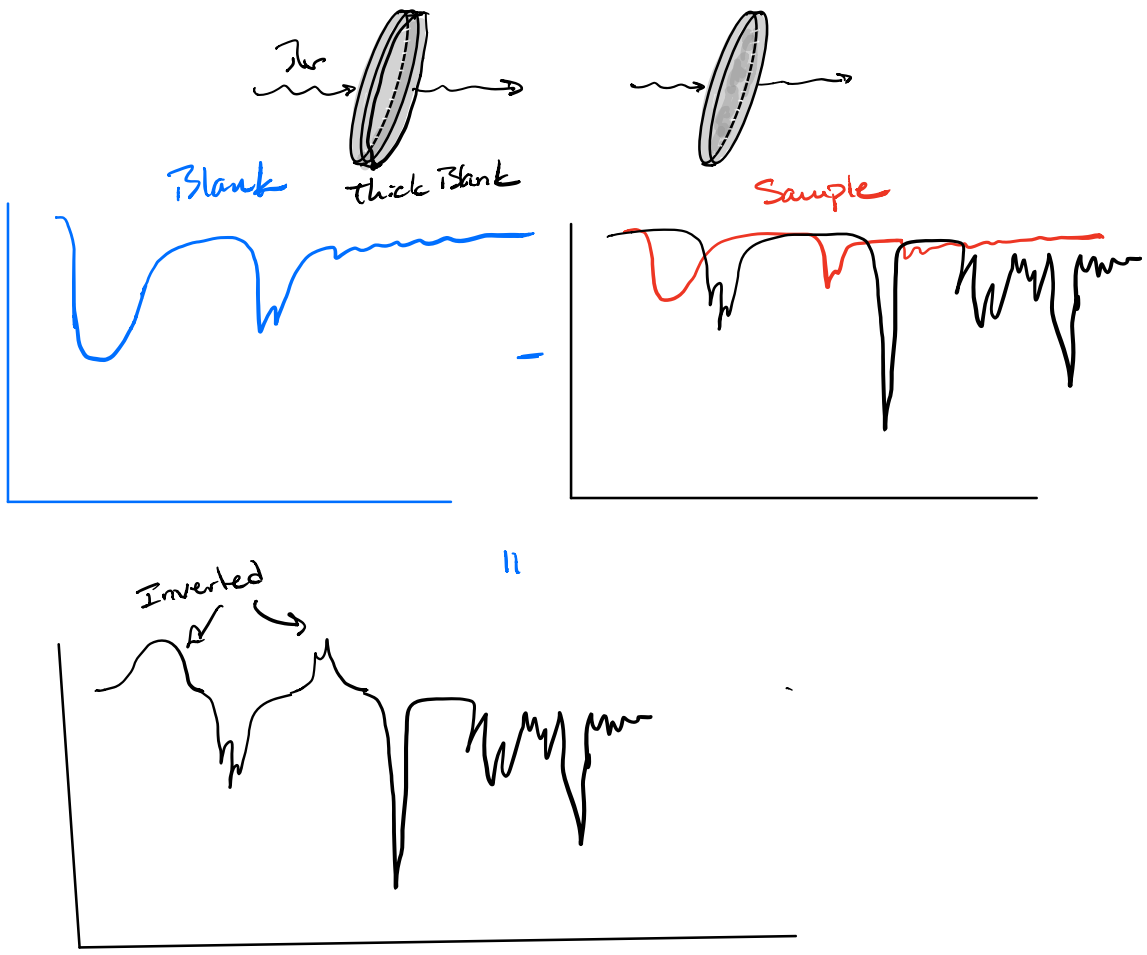
2400 cm^{-1}

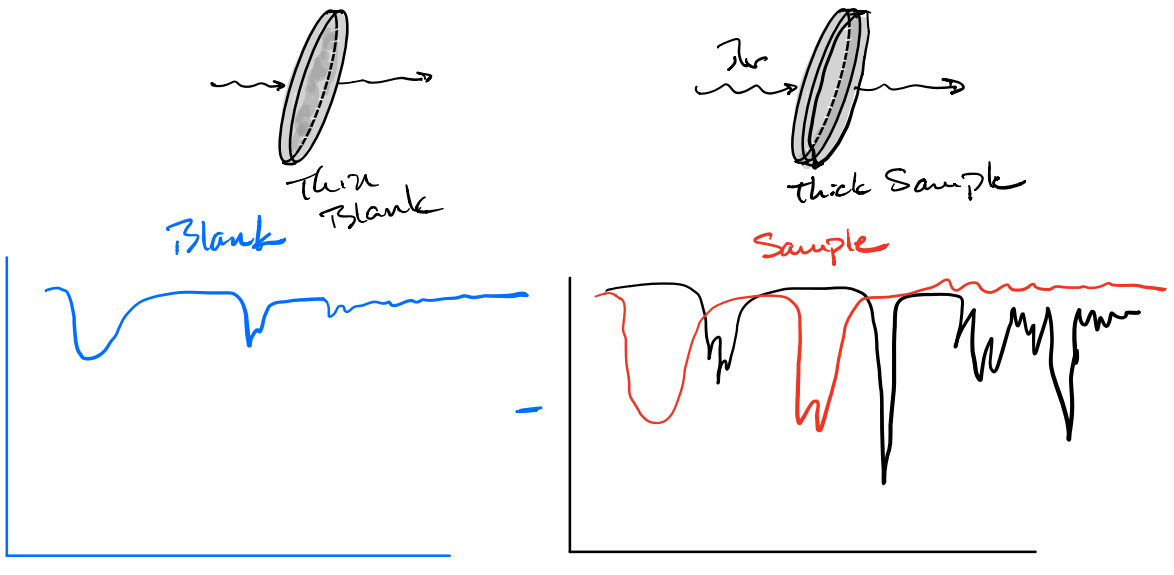
Blank w/ same path length as sample

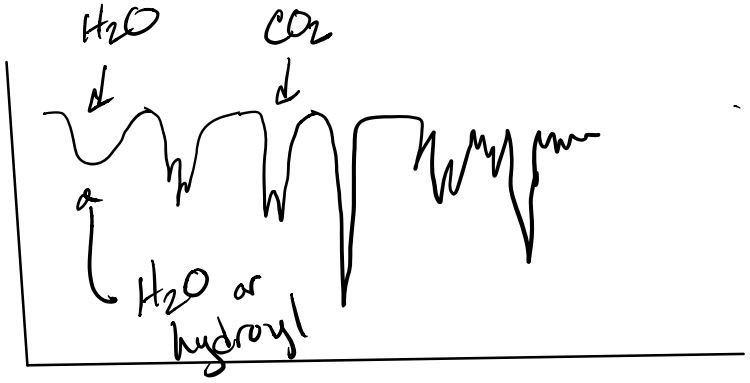


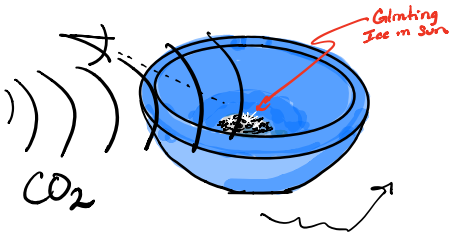
=




Blank
Thick Blank
Sample
Inverted


Thin Blank
Thick Sample
Blank
Sample


H_2O
CO_2
H_2O or hydroxyl

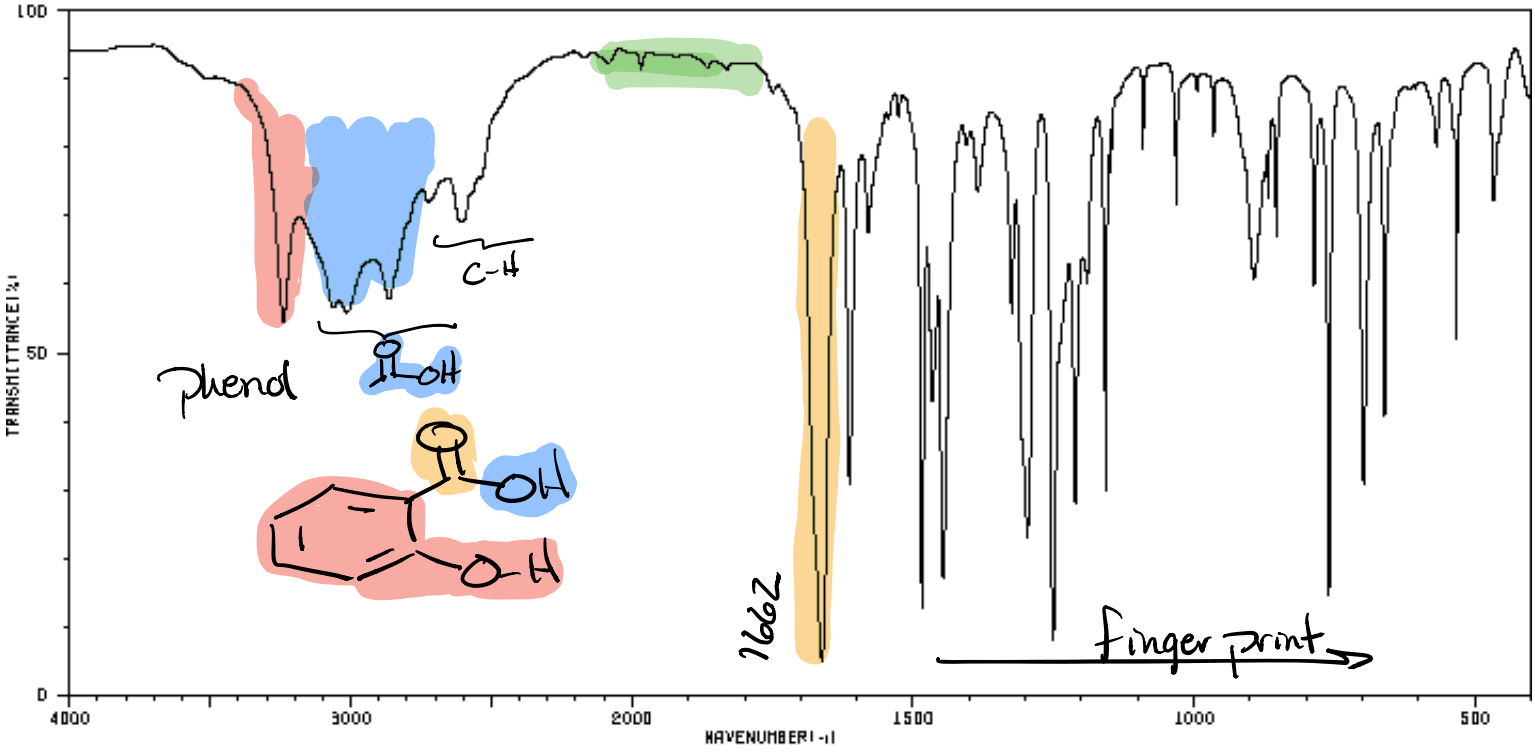


KBr absorbs CO_2

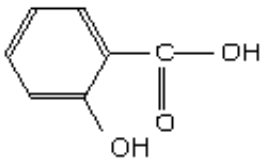
HIT-NO=1330	SCORE= ( )	SDBS-NO=1149	IR-NIDA-08453 : KBR DISC

SALICYLIC ACID

$C_7H_6O_3$



3240	62	1680	64	1326	63	1161	72	786	67
3013	53	1527	81	1297	21	1091	77	760	13
2864	55	1484	12	1251	7	1032	68	699	29
2724	70	1467	41	1239	49	966	79	661	38
2605	66	1447	16	1212	26	893	58	589	77
1662	4	1405	77	1190	58	868	70	533	50
1613	29	1386	70	1167	28	853	64	467	70

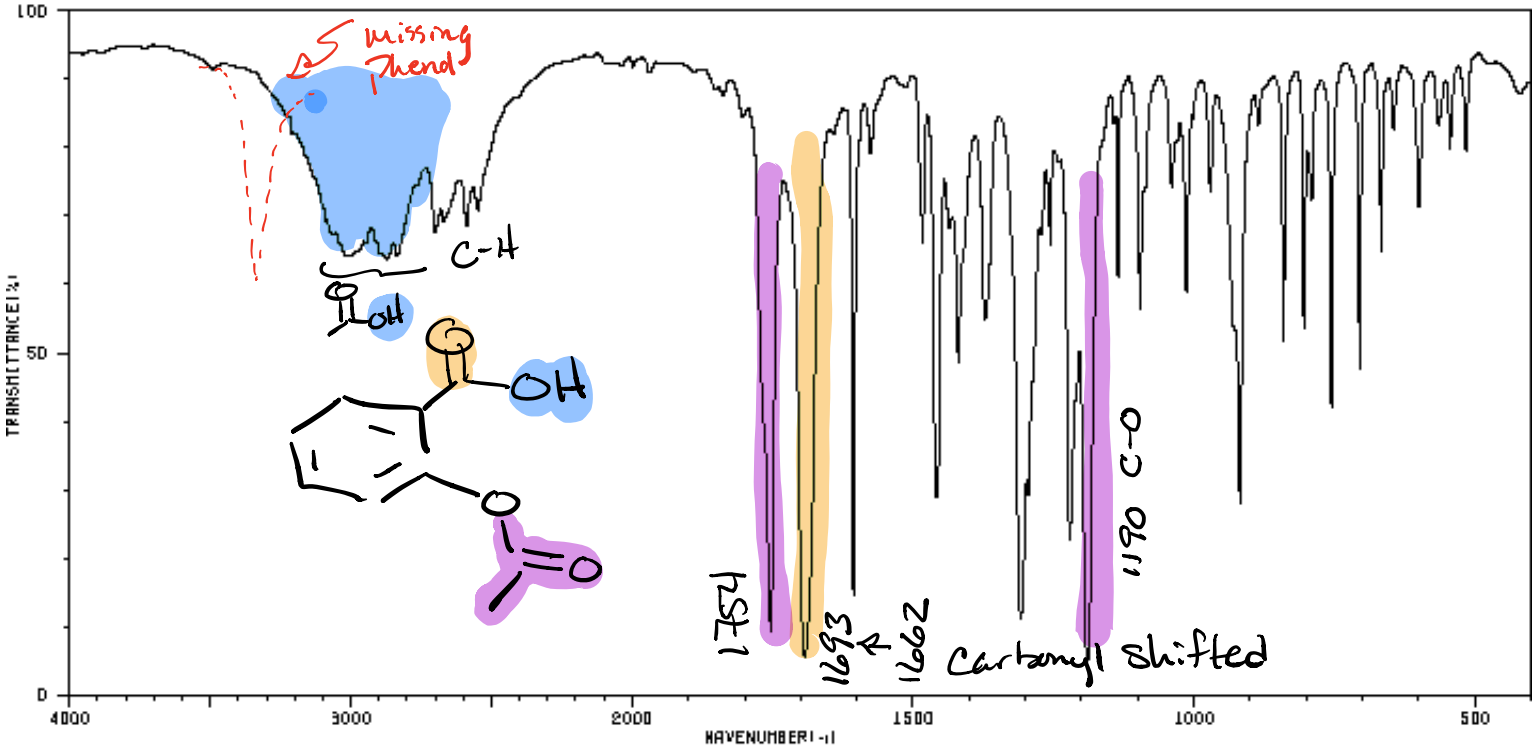


HIT-NO=1022	SCORE= ( )	SDBS-NO=532	IR-NIDA-63853 : KBR DISC

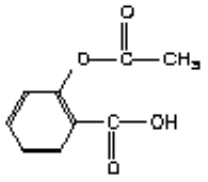
O-ACETOXYBENZOIC ACID

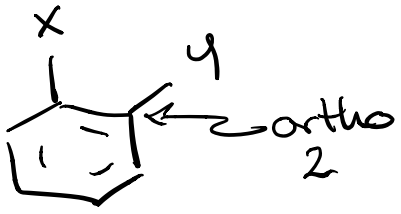
ortho-

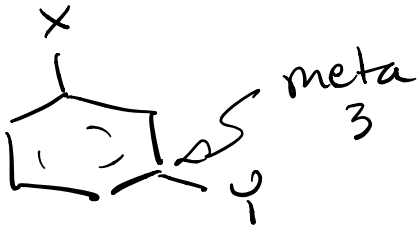
$C_9H_8O_4$

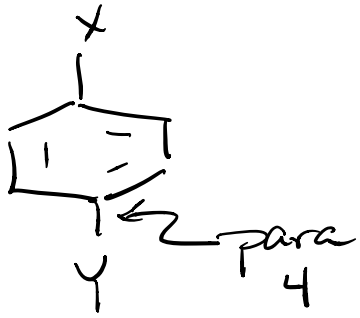


3006	62	2689	66	1436	66	1222	21	918	26
2983	62	2548	68	1420	47	1190	4	841	50
2891	62	1754	9	1372	53	1136	58	805	52
2872	62	1693	6	1308	10	1096	66	766	41
2834	62	1606	14	1295	28	1014	57	706	46
2701	66	1483	64	1272	64	971	70	667	62
2670	66	1469	27	1257	64	928	62	600	68







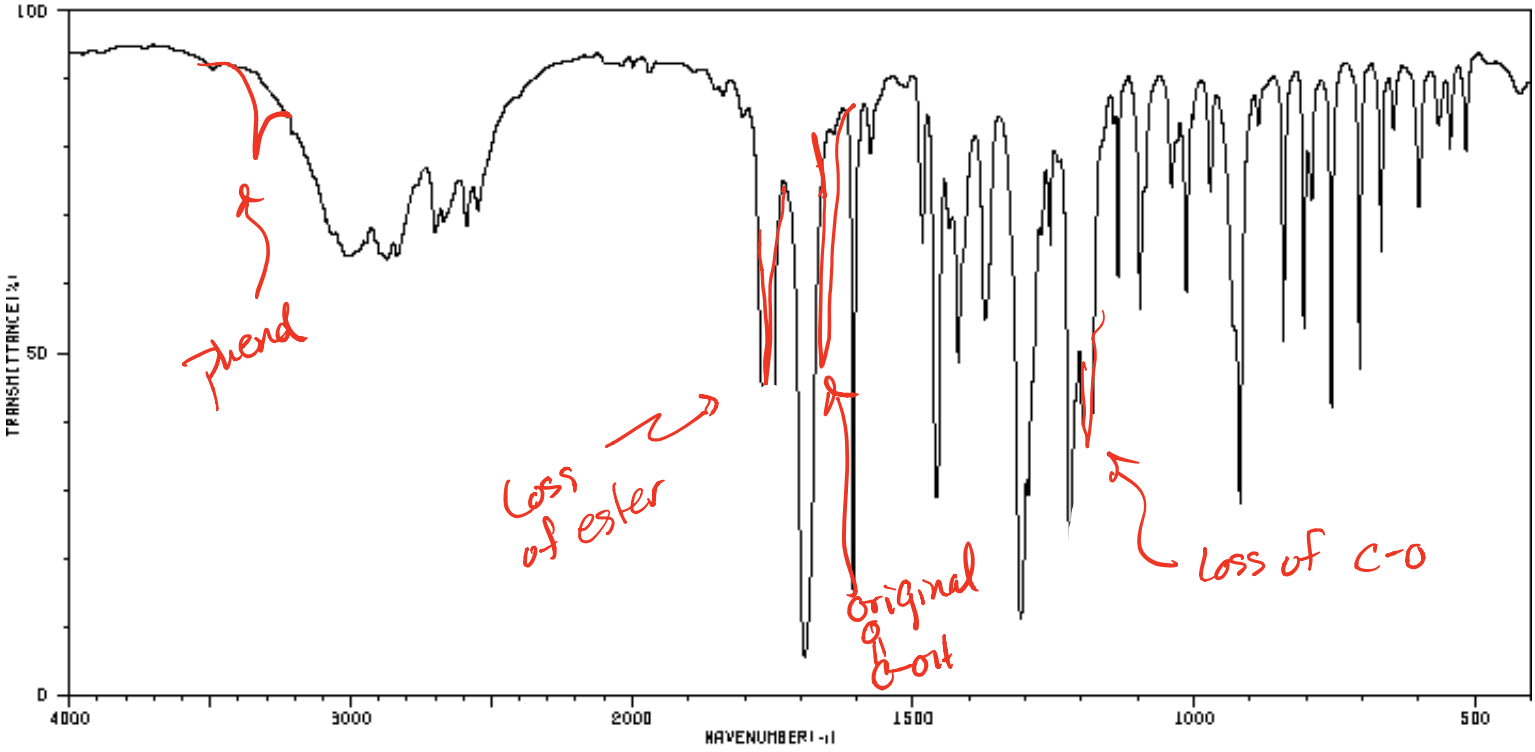


HIT-NO=1022	SCORE= ( )	SDBS-NO=532	IR-NIDA-63853 : KBR DISC

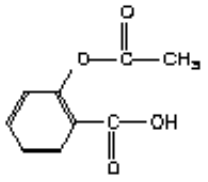
O-ACETOXYBENZOIC ACID

If Contaminated with starting material

$C_9H_8O_4$



3006	62	2689	66	1436	66	1222	21	918	26
2983	62	2548	68	1420	47	1190	4	841	50
2891	82	1754	9	1372	53	1136	58	805	52
2872	62	1693	5	1308	10	1096	66	766	41
2834	62	1606	14	1295	28	1014	57	706	46
2701	86	1483	64	1272	84	971	70	667	62
2670	66	1469	27	1257	64	928	62	600	68



2 Solubility
3 Crystallizations
4 Extraction

} Lab notes
Questions from
Pavia → Question
Section
&
Report Section

5 Chromatography nothing due
6 Distillation nothing due
7 IR Spectroscopy nothing due
8 Synthesis of Acetylsalicylic acid * writing
assignment