Lab #5
Preparation of Benzoic Acid using a Grignard Reagent

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CHM1321 section A3
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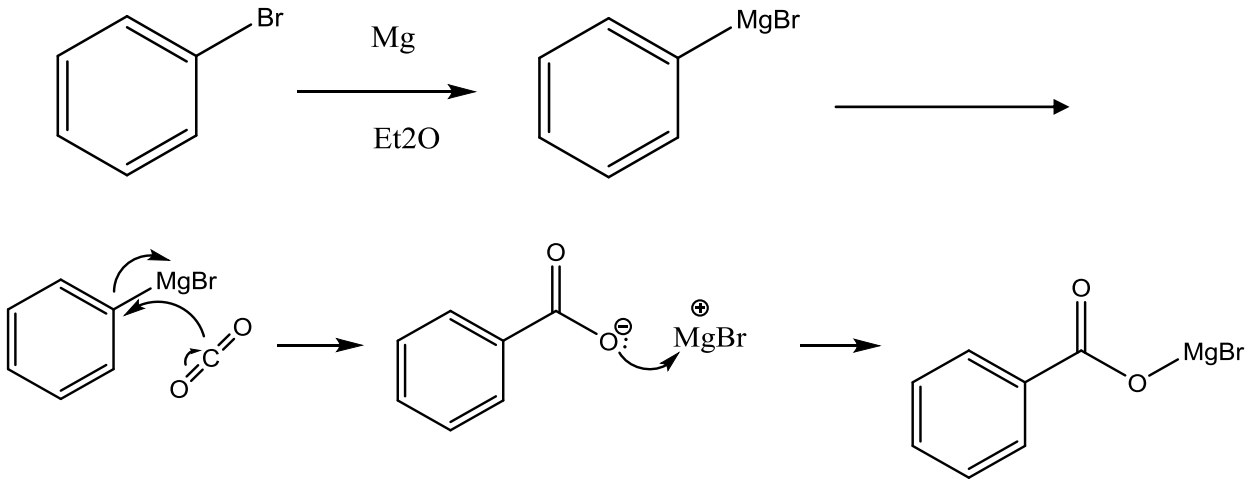
March 1st, 2016

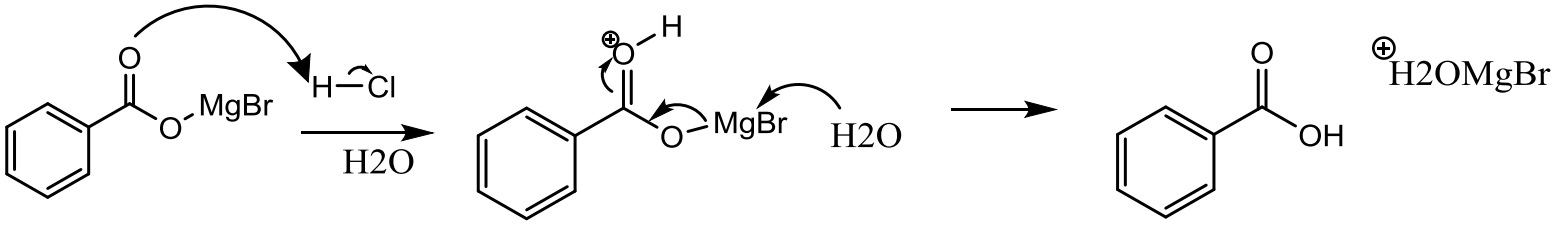
Procedure: see pages 43-47 of lab manual, Rashmi Venkateswaran, *Preparation of benzoic acid using a Grignard reagent,* Experiment No. 5, 2016, pp43-47.

Observations:

Mechanism 1: reaction

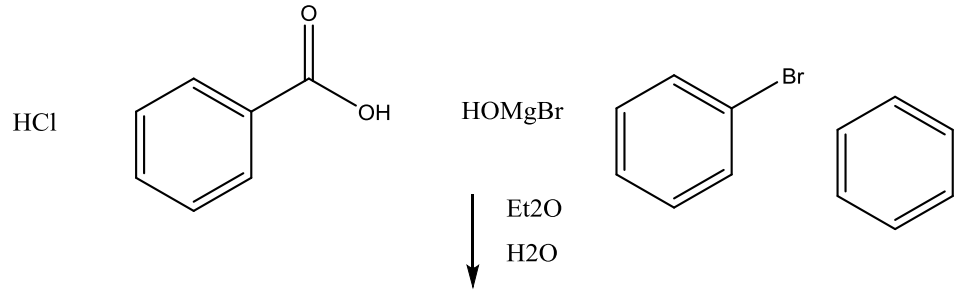


Mechanism 2: workup



Flowchart: Extraction

First separation occurs with extraction of the aqueous layer on the bottom which in this case is water. Aqueous layer contains HCl and HOMgBr which are both polar. Diethyl ether is the organic layer and will contain bromobenzene, benzoic acid and some benzene. Next, water and NaOH are added to remove the acidic hydrogen from the benzoic acid. A final separation using diethyl ether and water occurs to move the now polar benzoic acid into the aqueous layer and the non polar benzene and bromobenzene into the organic layer. HCl is added to put the H back onto the benzoic acid. Water is filtered out to get pure solid benzoic acid.

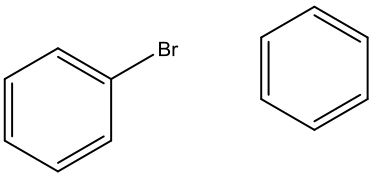


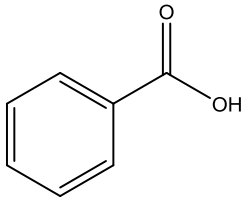
H2O

Et2O

HCl

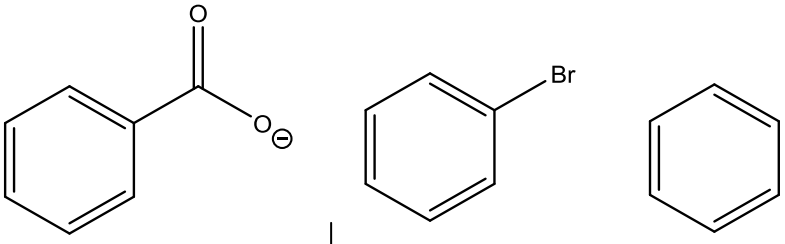
HoMgBr





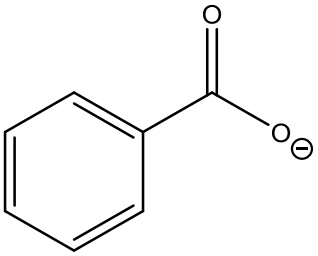
H2O

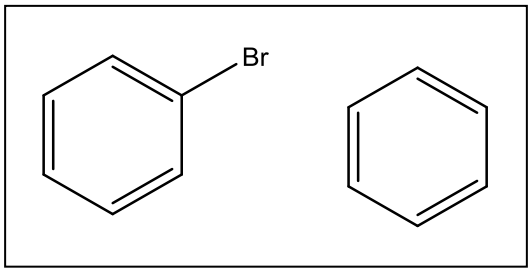
NaOH 10%



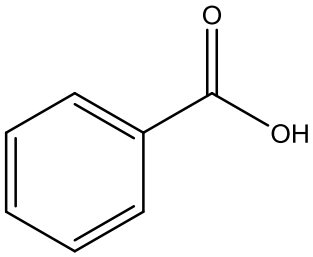
H2O

Et2O





HCl



(solid)

Filter off H2O

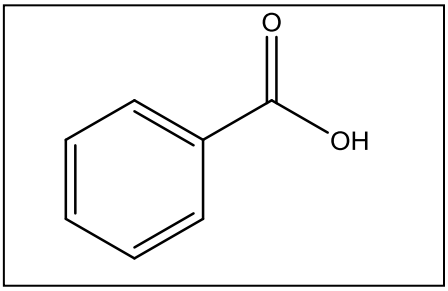
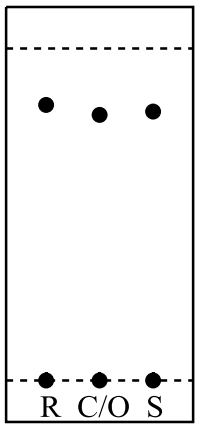


Table of reagents:

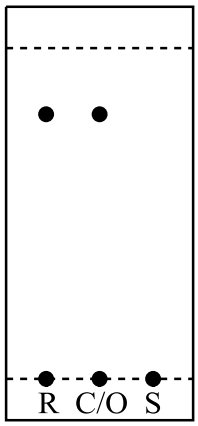
Compound	Molecular weight	Amount	Moles	Density
Magnesium	24.31 g/mol	0.81 g	0.033 mol	-
Bromobenzene	157.01 g/mol	3.0 ml	28.66 mmol	1.5 g/cm^3
Iodine	126.90 g/mol	1-2 crystals	-	-
Dry ice (CO_2)	44.01 g/mol	66.67 ml	1.5 mol	1.5 g/cm^3

TLC 1:



Compound	Rf value
R: Bromobenzene	0; 0.83
C/O: co-spot	0; 0.80
S: Organic layer	0; 0.81

TLC 2:



Compound	Rf value
R: Bromobenzene	0; 0.80
C/O: co-spot	0; 0.80
S: Dry product	0

Observations:

- Initial mixture first is reddish in colour, intense bubbling starts, mixture turns brown
- Bubbles stopped, then restarted upon addition of second half of mixture being added
- brown colour remained after 30 minutes + the water bath
- addition of dry ice formed a cold, sticky solid the consistency of ice cream
- addition of remaining reagents caused mixture to separate into clear aqueous layer and yellow organic layer

Percent yield:
molecular weight of benzoic acid: 122.22 g/mol
moles of bromobenzene: 28.66 mmol
28.66 mmol x 122.22 g/mol= 3.5 g
experimental yield: 4.46 g
(4.46/3.5) x 100%= 127.4 %

Discussion:

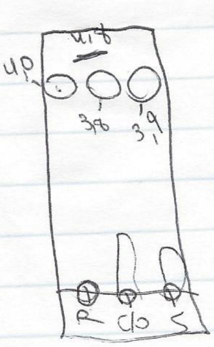
- % yield is over 100% because too much HCl was added, causing mixture to salt out and NaCl to form, was also not completely dry when measured.
- melting point obtained was 114.0 ℃, product appeared as white solid, a little sticky because not completely dry.
- Iodine reacts with surface of magnesium to expose fresh magnesium for the Grignard
- Initial reaction is between bromobenzene and magnesium to make Grignard
- Next reaction is between CO_2 and Grignard to get R-OMgBr
- Final reaction involves water and HCl which breaks the bond between the O and the MgBr which will bond with the water. The HCl will add an H to the benzoic acid, giving a final product.
- Water cannot be present in grignard reaction because it will protonate and "kill" the reaction
- Carbon dioxide is present in the form of dry ice
- Rf values for the second and final TLC plate show that bromobenzene is not present in the product which shows that benzoic acid was successfully isolated
- $CaCl_2$ used in drying tube because it reacts with water in the air to keep the water out of the reaction

Questions:

1. Anhydrous Ether is used in the reaction because water will protonate the Grignard and kill the reaction.
2. Water bath is used to heat the reaction because anhydrous ether is extremely flammable and it is dangerous to have electricity around it.
3. Dry ice is solid carbon dioxide, C=O=C.
4. If the dry ice is not freshly obtained it will sublime and you will be using water in your reaction instead.

2 mechanisms: 1 for reaction, 1 for separation
flow chart
reagent table

Part A:
~ 20 ml diethyl ether
3.0 ml bromobenzene
0.81 g Mg
1-2 crystals iodine
= intense bubbling, mixture is brown
- Started off reddish, soon disappeared
- Upon addition of second half, bubbles recommenced
- brown color stayed
- After 30 minutes + water bath,
- Addition of dry ice formed product consistency of ice cream
- Addition of multiple other products caused mixture to separate into yellow organic layer and clear aqueous layer



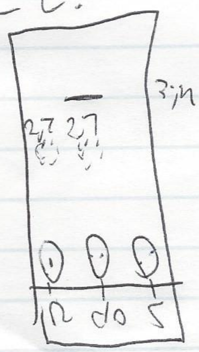
R = Bromobenzene
C/S = cospot
S = organic layer

- Too much HCl added, NaCl precipitated out into product

- watch glass + filter paper = 31,14 g
  " " " " + product = 35,68 g

melting point = 114,0 °C

TLC 2:



R = Bromobenzene

co = cospot

S = dry product