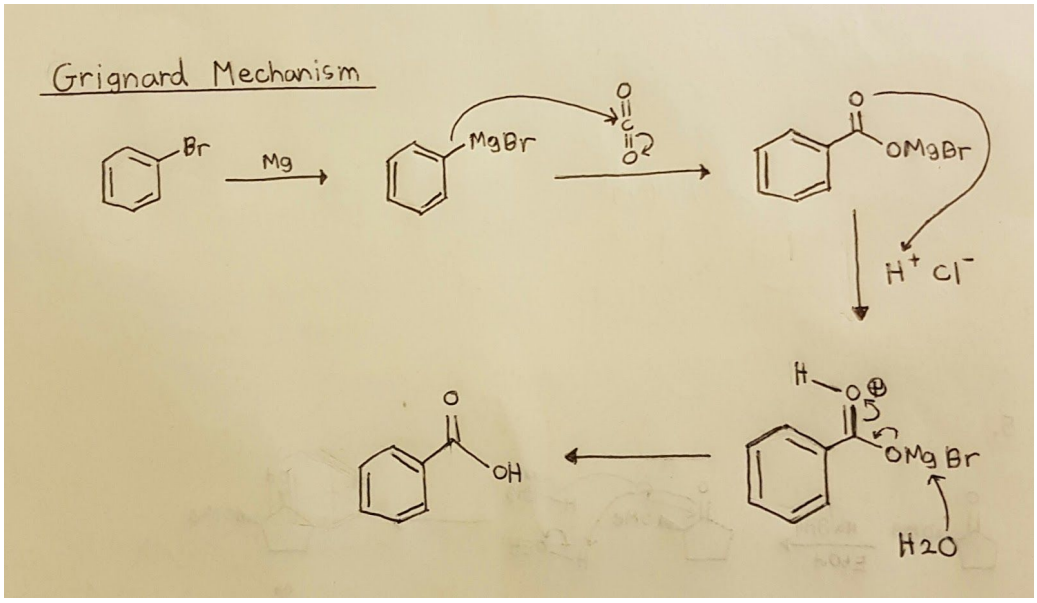
Experiment #5: Preparation of Benzoic Acid using Grignard Reagent

Introduction

Grignard Mechanism of the reaction



Procedure

"As described in the lab manual (Experiment #5, Pg. 43-47, author: Title: CHM 1321 Introductory Organic Chemistry Laboratory Manual 2017)"

Table of reagents

Reagent	Amount (g/ml)	Molec. Weight (g/mol)	# mol	Density (g/ml)
Mg	0.8 g	24.31	0.0329	-
Bromobenzene	3.0 ml	157.01	0.0287	1.5
$(C_2H_5)_2O$	20 ml	74.14	-	-

Observations

- Magnesium turnings were a rustic grey colour
- Iodine crystals were black obsidian rock shards
- $CaCl_2$ were solid round spherical white balls
- Anhydrous Diethyl Ether was a colourless liquid
- Mixture had a lime coloured ether layer at the top and a layer of colourless solution at the bottom

Table of results

Product	Amount (g/ml)	Molec. Weight (g/mol)	# mol	% yield	Melting point (ºC)
Benzoic Acid	2.87 g	122.12	0.0235	82%	127

TLC

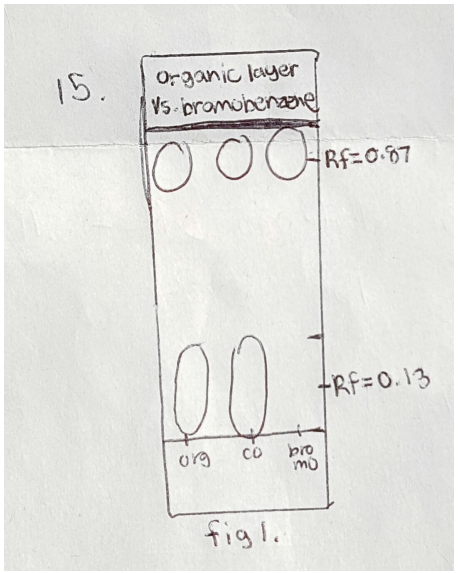


fig 1.

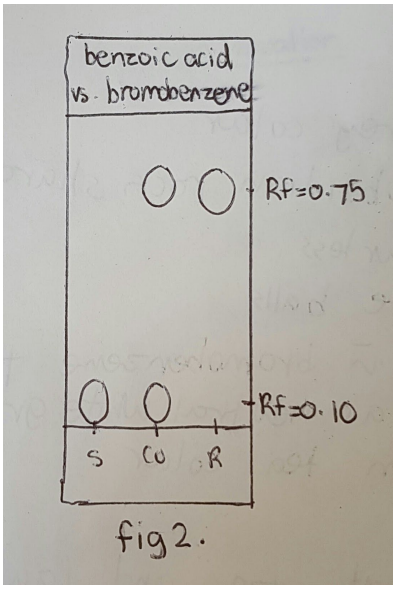
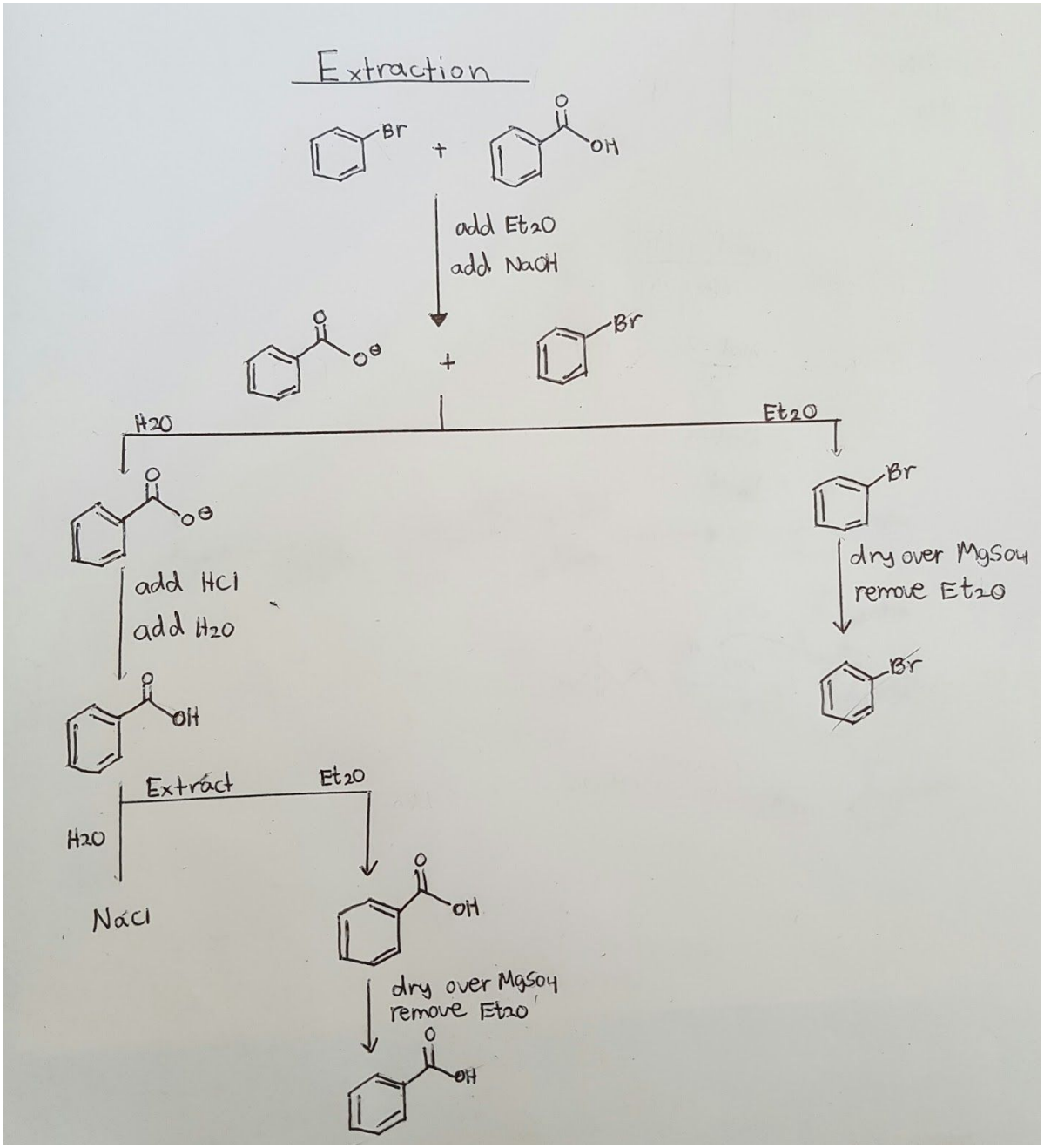


fig 2.

Flow chart



Calculations

Amount of Bromobenzene = 0.0287 mol
Amount of Benzoic acid = 0.0235 mol

Yield % = (# mol of product / # mol of starting material) x 100
= (0.0235 mol / 0.0287 mol) x 100
= 81.9% = 82%

Discussion

This experiment involves the extraction between carboxylic acid and impurities such as bromobenzene. This process is initialized by first mixing bromobenzene in Iodine activated magnesium. This causes a Grignard mechanism and the formation of a carboxylic acid once extracted using an organic solvent of Diethyl Ether, NaOH, and HCL. The reason why Anhydrous Diethyl Ether is used is because if moisture is included in the Grignard reaction then a side reaction of benzene would be produced in the reaction which would cause the addition of an impurity in the product which is not what is needed. To reduce moisture in the reaction, a drying tube consisting of $CaCl_2$ and an oven heated apparatus was used for the grignard reaction. The figure 1. TLC proves that the organic layer has some bromobenzene however the figure 2. TLC shows the sample has no bromobenzene which proves the reaction went to completion and benzoic acid has been produced successfully. The product came out as a solid white powder which was measured to have a melting point of 127 ^{o}C. Benzoic acid originally has a melting point of 122 ^{o}C which means the product is the right product however there are most likely some impurities in the product. Some sources of error that could have caused the formation of impurities could be perhaps keeping the heated apparatus open to collect moisture from the atmosphere, The dry ice could have been exposed too long causing the condensation of water on the ice which would introduce moisture into the reaction and thus producing some benzene impurity, or the stirring of the magnesium did not go to completion because the stirring time was cut short causing the leftover of the bromobenzene impurity.

Questions

1. Anhydrous ether is used in the reaction because if moisture is included in the Grignard reaction then a side reaction of benzene production in the reaction would cause the addition of an impurity in the product which is not what is needed.

2. A hot water bath is used in the grignard reaction to heat the sample in order to continue the reflux of the reaction so the vapours of the system recondense back into the original system to insure all the product is claimed and the yield is as close as possible to 100%.

3. Dry ice is frozen carbon dioxide which has a surface temperature of -78.5 ^{o}C. Instead of melting to a liquid it undergoes sublimation to produce vapours.

4. Freshly obtained dry ice is important because if dry ice is exposed to the atmosphere then water will condense at the surface which would produce the impurity benzene in the grignard reaction thus making the product less pure.