

Student Name: Elodie Howard-Maurice

Student Number: 8241985

Partner's Name and Student #: Haneen Abu-Dakka 8251768

Lab TA Name: Parsa Jamshidi

Lab Day: Tuesday morning

Lab Week: 2

Laboratory Report

Experiment 5.

March 29th 2016

Preparation of Benzoic Acid using a Grignard Reagent

Introduction

Grignard Reagents are prepared and used in this experiment in order to produce benzoic acid. These reagents represent one of the first organometallic reagents and are useful in forming bonds to carbon atoms. The general structure of a Grignard Reagent is of the form RMgX where R represents either an alkyl group and X is used in representing the halogen like bromine. During the reaction there is a definite reaction between magnesium and the halogen. In terms of Grignard Reagents the most important are the nucleophilic compounds especially those that contain polarized double bonds (aldehydes, ketones, and esters) which then form alcohols. During the reaction between an aldehyde and a ketone the pi bond is displaced off the carbonyl and reacts with the Grignard Reagent, this causes formation of an alkoxide. The alkoxide further interacts with the magnesium ion forming a magnesium alkoxide. During this experiment, Bromobenzene is treated with Magnesium in a diethyl ether solution creating a Grignard reagent. The reagent is reacted with CO_2 and hydrolyzed with an acid. Once this solution is obtained, the impurities are washed out with a separatory funnel; the remaining solution is treated until a precipitate is formed. This precipitate is extracted using suction filtration and benzoic acid is isolated.

Procedure

Refer to Organic Chemistry lab manual pages 46-47. No changes have been made.

Observations

Table 1. Table of Reagents Part A

Compound	Amount (g or ml)	Molecular Weight (g/mol)	Density (g/cm^3)	Moles (mol)
Iodine	0.09	125.90	4.93	0.0007
Magnesium Turnings	0.8 g	24.31	1.74	0.03
Ordinary diethyl ether	15 ml	74.12	0.713	0.20
Anhydrous diethyl ether	20 ml	74.12	0.713	0.27
Bromobenzene	3 ml	157.01	1.50	0.019

Note that molecular weight and density values were searched up on the internet. Amount was obtained during the lab. Moles were calculated from the data in the table.

Observations

- Reaction mixture went from a dark rusty red to yellow then a very light cloudy white color
- I was able to see the Mg (magnesium turnings) at the bottom of the solution in the round bottom flask
- The reaction is working very slowly, it barely started to boil and when it did, the boiling was very not very visible.
- When reaction flask was put into the hot water bath, the solution turned into a brown turbid(cloudy) color
- When the solution was added to the dry ice, gas escaped the beaker. When adding ice, water, HCl, etc. to that solution is reacted violently and created a thick sticky white solid.
- As the solution was melting it created 2 layers, a yellow & a white which must have been the organic and aqueous phases
- The ice took a while to completely mix and dissolve the dry ice
- The precipitate looked white and lumpy

Calculations

Moles benzoic acid = Moles Bromobenzene (1:1 ratio)

Theoretical yield of benzoic acid

$$\begin{aligned}m_{\text{benzoic acid}} &= n_{\text{benzoic acid}} \times M_{\text{benzoic acid}} \\ &= (0.019 \text{ mol}) \times (122.12 \text{ g/mol}) \\ &= 2.32 \text{ g}\end{aligned}$$

$$\begin{aligned}\% \text{ Yield}_{\text{benzoic acid}} &= (\text{experimental yield} / \text{theoretical yield}) \times 100 \% \\ &= (0.2 \text{ g} / 2.32 \text{ g}) \times 100\% \\ &= 8.6\%\end{aligned}$$

Discussion

Preparation of Grignard Reagent. In the first step of the procedure all the glassware was cleaned and dried to ensure that all equipment was free of water, because during a Grignard Reaction the presence of water will cause the protonation of the Grignard Reagent and therefore making it very difficult to form the desired magnesium intermediate. If the magnesium intermediate does not form, magnesium will precipitate out and the reaction will turn to a milky white solution indicating that the reaction mixture has been contaminated with water and can no longer continue on. We then fill the drying tube with CaCl₂ before the glass cools in order to prevent water from entering the reflux apparatus. The CaCl₂ is used because it will react with water in the air thus preventing water from

entering the apparatus. Next, we prepare the Grignard Reagent using 0.8 g of magnesium turnings two iodine crystals (0.09g). Iodine is used in order to oxidize the magnesium from Mg^0 to Mg^{2+} . Then, bromobenzene and anhydrous diethyl ether is mixed with the solution. The iodine degrades the oxide layer that is present on the magnesium turnings. The reddish color of the I_2 starts to disappear as it has started to react with the magnesium to form MgI_2 which removes any excess water from the apparatus. Although our solution didn't boil very much, it was still able to go through this whole process. Putting the reaction mixture into a hot water bath enhanced the boiling and also ensured that the reaction achieved or closely achieved completion. The final mixture is phenylmagnesium bromide

Reaction with carbon dioxide. The phenylmagnesium bromide in diethyl ether precipitated an undissolved residue, once it settled we added dry ice to the Grignard Reagent. Adding dry ice to the Grignard Reagent caused the carbon dioxide from the dry ice to react with the carbonyl functional group on the Grignard Reagent. With this reaction the Grignard Reagent has an extra carbon added to it causing the formation of a carboxylic acid.

Isolation of benzoic acid. Ice was added to ensure that the solution was cooled down. HCl was used in order to protonate the oxygen after the $MgBr$ was removed by the addition of water and ice water. The diethyl was used to remove any anhydrous diethyl ether from the aqueous layer and making separation of the two layers easier. Water has a higher density than the organic layer so therefore it rests at the bottom of the solution. The aqueous layer is extracted again with 10% NaOH in order to neutralize the acid. HCl is re-added to the aqueous solution and all that is left in the solution is benzoic acid. We place it in an ice bath in order to allow the solution to cool and for the precipitate to form. The solid product was separated from the solution using suction filtration. The final product was then put into the melting point apparatus in order to determine its melting point. The melting point of 110 degrees Celsius obtained.

Sources of error. There could have been many things that contributed to the low yield that we obtained. Since this experiment is so sensitive to water there could have easily been small water contaminations that gave us less of a yield. The iodine crystals react with magnesium (Mg) so too much iodine could have contributed to the low yield. Lastly the transferring of solutions from glassware to glassware, not everything is measured accurately and not every single last drop of solution gets added to the reaction mixture.

Questions

1. Anhydrous ether is used in this reaction because it does not contain any water and therefore water cannot readily react with the Grignard Reagent. Water causes protonation

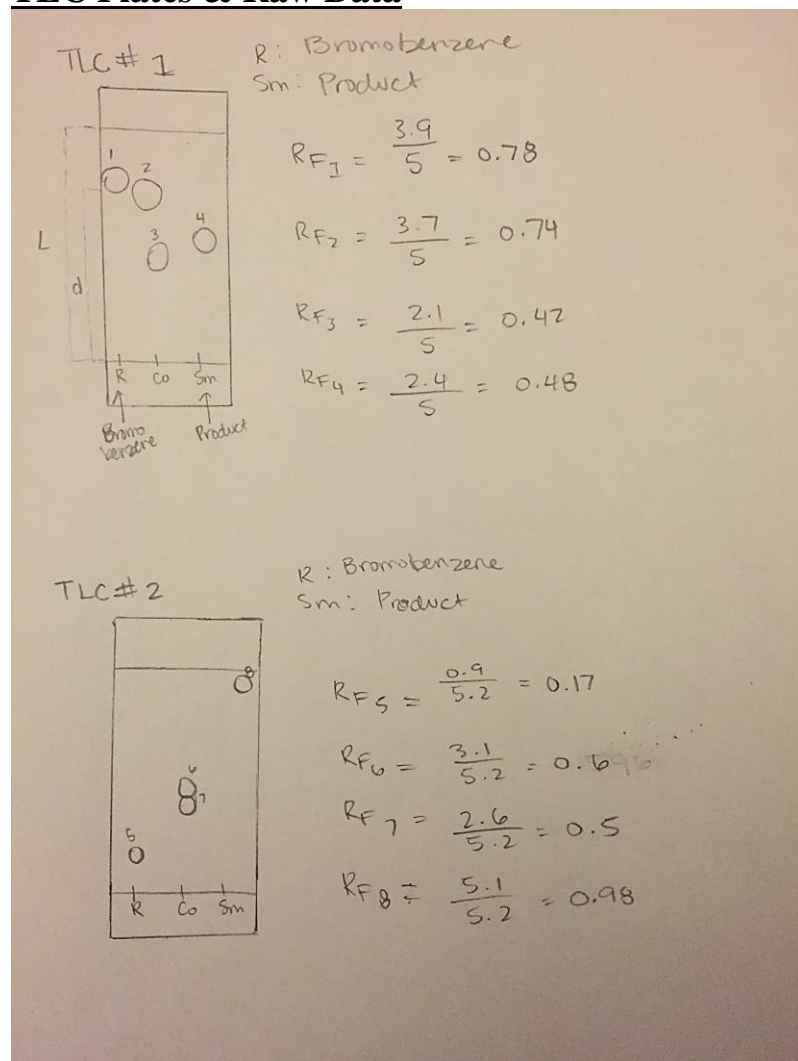
and makes the Grignard reagent inactive. Water will cause an acid-base reaction and not a carbonyl reaction.

2. A water bath is used to heat the reaction because it helps push the reaction to completion. Ether has a low boiling point of 37°C so the increase in temperature allowed the solution containing the ether to continue taking place.

3. Dry ice is the solid form of carbon dioxide with a temperature of -78.5°C . It is called "dry ice" because it sublimates directly from a solid to a gas.

4. In this experiment it is important to freshly obtain dry ice because dry ice is extremely cold and the condensation of moisture will reduce the yield of the product. The water will react with the Grignard reagent and destroy it by producing a completely different reaction.

TLC Plates & Raw Data



Experiment #5

0.80g of magnesium turnings

3ml Bromobenzene

20ml diethyl ether

2 crystals iodine \rightarrow 0.09g

Product 0.2 g

- Mixture went from a dark rusty red to yellow and then very cloudy white color

- Able to see the Mg ^{light} @ bottom of the round bottom flask

- Mg rxn is working very slowly

- ~~Solution seems to be clearing up~~

- Boiling in hot water bath and brown turbid (cloudy) solution but solution did not ~~completely~~ boil on its own

- When adding ice, HCl, etc to dry ice, it steamed?? created a sticky solid then as it was "melting" created 2 layers yellow & white. Vident rxn

- This took a while to completely mix & dissolve dry ice

- After both extractions the addition of HCl to aqueous phase heated up the solution

- Precipitate was dry & looked white & powdery

- Melting point of product 110°C