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**Grignard Reaction**

**Purpose:** The purpose of this experiment is to further our understanding and conduct a reaction known as the Grignard Reaction. Through this reaction, we are able to synthesize a tertiary alcohol by using an excess of an organomagnesium halide such as phenylmagnesium bromide.

**Reaction(s):**



**Physical Properties of Reagents:**

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Reagent | Structure | Molecular Formula | Molecular Weight (g/mol) | Boiling Point (°C) | Density  (g/mL) |
| Bromobenzene |  | C6H5Br | 157 | 156 | 1.5 |
| Methyl benzoate |  | C8H8O2 | 136.15 | 199 | 1.08 |
| Diethyl ether |  | (C2H5)2O | 74.12 | 94.28 | 0.71 |
| Dichloromethane |  | CH2Cl2 | 84.93 | 103.3 | 1.33 |
| Hexane |  | C6H14 | 86.18 | 68.0 | 0.655 |

**Procedure:**

Start by gathering the necessary lab materials and dressing in the proper lab attire. Next, begin by forming the Grignard reagent. First, measure and add 4.2 mmol of magnesium shavings into a 10 mL round-bottom flask with a spin vane, 3mL of diethyl ether, and a few crystals of iodine. You will place this over a hot plate with the heat off but utilizing the spin feature. You will assemble the apparatus from the figure provided from the handout. Be sure to close off the open end of the apparatus with a rubber septum to ensure that no product is lost in the process.



Measure and add 5.0 mmol of bromobenzene using an analytical balance into a 5 mL conical vial followed by 1.5 mL of diethyl ether. Next, you will carefully utilize a 3mL syringe and completely extract the newly made solution. Insert the syringe containing the solution through the rubber septum. Over the course of 30 minutes carefully and slowly add the solution into the 10 mL round bottom flask. During this time, the temperature of the hot plate should be set at 2. Allow the reflux to continue until all the magnesium shavings have completely dissolved. Over the course of the experiment be sure to document any noticeable observations.

Next, conduct the final formation of the product, triphenylmethanol. Do this by placing 2.0 mmol of methyl benzoate into a 3.0 mL conical vial followed by 1.5 mL of diethyl ether. Using an additional 3.0 mL syringe, carefully and completely extract the solution. Insert the syringe into the rubber stopper and add the solution into the 10 mL round bottom over the course of 5 minutes. After this has been completed, you will allow the solution to reflux for a total of 30 minutes. Be sure to record any observations during this time. After the reflux has been completed, allow the solution to cool to room temperature by removing it from the hot plate. Next, add 20 drops of water to the round bottom flask under the fume hood. Additionally, add 3M HCl using a micropipette to the flask until no more bubbles emerge from the solution. Be sure to spin continuously over a hot plate without the heat function. During this time, one will notice the emergence of the aqueous and organic layer.

Next, it is time to perform the extraction of the product. Carefully remove the spin vane and begin by transferring the solution from the 10mL round bottom flask into the separatory funnel. Using a 10 mL graduated cylinder, add 10 mL of distilled water and 10mL of diethyl ether to the separatory funnel. Place a glass stopper on the top of the separatory. Firmly and securely mix the solution within the funnel, and vent the trapped gas. Return it to its upright position and allow the layers to separate. Remove the aqueous layer by collecting it in a 100 mL beaker. Next, wash the remaining organic layer with 10 mL of water and 10 mL of NaCl using a graduated cylinder. Collect the remaining organic layer in a 50 mL Erlenmeyer flask and add sodium sulfate until it no longer clumps. Transfer the solution into an additional 50mL Erlenmeyer flask with a boiling chip. Use a hot plate under the fume hood to boil off the excess solution and collect the precipitate. Perform the recrystallization process by using dichloromethane to completely dissolve the participates collected in the previous step. Next add roughly four times the amount of hexane. Heat under the fume hood until the solution clears up. Allow the solution to cool and observe as the solution begins to crystalize. Be sure not to move or disturb the solution too much during this process. Use an ice bath to help the crystallization process. Set up the vacuum filtration apparatus and pre-wet the filter paper with hexane. Use vacuum filtration to collect and dry the crystals. Dry the crystals on the Hirsch funnel for 5 minutes. After you have completed this, transfer as much of the product onto a watch glass that has been pre-weighed.

To perform the melting point, use a capillary tube and crush the crystals into a powder. Tap the capillary tube onto the powder until roughly 3-5 cm has entered the tube. Utilize the melting point apparatus by setting a plateau and watching as the inserted capillary tube containing the compound melts under a given temperature. Perform the IR first by creating the necessary KBr pellets used to conduct the reading. Mix a small sample of your recrystallized product with the KBr salt. Next compress a small amount of the new mixture into a flat disk and conduct the IR reading. To perform the NMR reading, mix .700 g of dichloromethane with 10mg of the product. Add this mixture into a clean NMR tube and perform the reading.

**Observation:**

Beginning Mixture: Dark brown color

4 minutes into reflux: Faded from dark brown to light brown

6minutes 30 seconds into reflux: dark yellow-orange color

9 minutes 30 seconds into reflux: Dusty yellow color

30 minutes into reflux: Returned to dark drown color

55 minutes into reflux: No more magnesium present

Upon adding 3mL methyl benzoate/diethyl ether: turned reddish brown

7 minutes into addition of solution: turned dusty light orange color

**Data/Results:** See notes for calculations

* Theoretical Yield:
  + Start: Methyl Benzoate (Limiting reactant): 0.273g
  + Yield: Triphenylmethanol: 0.522 g
* Actual Yield:
  + Yield: Triphenylmethanol: 0.201 g
* Percent Yield
  + = 38.51%
* Experimental melting point
  + 162-165oC
* List absorption bands detected from IR spectrum
  + C(sp2)-H: 3150-3050 cm-1
  + OH: 3600-3400 cm-1
* NMR: See Attached for assigned peaks

**Discussion:**

Based on the results that we achieved through this experiment, we may conclude that we were able to run a successful experiment. While we were only able to receive a yield of 38.51% there are many variables to account errors or inconsistency for such yield. For one, the frequent transfer amongst the solutions into various beakers accounts for a significant loss of the product. Additionally, there was the inability to complexly remove all of the product from the filter paper after performing recrystallization. Additionally, any impurities present within the system may have resulted inside reactions which may have All of these account for various reasons as to why the yield was so low.

After analyzing our results, we can see that we were able to receive results in line with the expected outcome. After conducting the melting point and receiving a given value of 162-165oC, we are able to see that it is in line with the theoretical value with is 162oC. This means that the sample had relatively few impurities since the melting point did not deviate from the expected value. Additionally, after conducting the IR spectroscopy we can confirm the given structure of our compound. We are able to see this from the absorption band from 3400-3600 cm-1 signifying an alcohol. The IR spectrum is also telling that the Grignard Reaction went to completion from the C(sp2)-H absorption band from 3150-3050 cm-1. Finally, the NMR reading confirms the given structure by analyzing the given peaks of the hydrogen. We are able to see the downfield and up field electrons in relation to the alcohol.

I would improve this experiment by using a stock solution of the Grignard reagents instead of synthesizing our own products to limit errors as well impurities that may be introduced into the system through synthesizing our own reagents. This would just help to confirm the outcome of the reaction.