The Grignard Reaction – Synthesis of Triphenylmethanol

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Experiment 10
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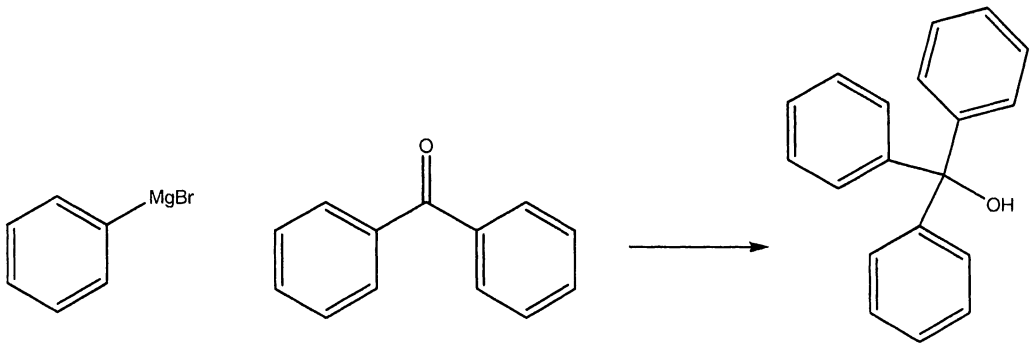
Experiment 10: The Grignard Reaction-Synthesis of Triphenylmethanol

Introduction

The purpose of this lab was to form triphenylmethanol using a Grignard reaction. The Grignard reactant was produced from Magnesium and Bromobenzene which was then reacted to benzophenone. The reaction was kept dry to enable the Grignard reagent to work properly. The formation of the final product, triphenylmethanol, was confirmed using a TLC of the product and of triphenylmethanol and an IR.

Reaction



phenylmagnesium bromide benzophenone triphenylmethanol

Table

Compound	Molecular Weight g/mL	Amount Used	mmol	Equivalents
Magnesium	24.31	0.050 g	2	1
Anhydrous Diethyl Ether	74.1224	1.2 mL		
Bromobenzene	157.0095	0.330 g	2.1	~1
Benzophenone	18.2	0.364 g	2	1
Anhydrous Ether		1 mL		
3M HCl	36.46	2 mL		
Petroleum Ether	87-90	0.5 mL		
NaCl	58.44			
$CaCl_2$	110.986			
Triphenylmethanol	260.32	0.521 g	2	1

Procedure

0.050g Mg powder was placed in a dry reaction tube capped by a cap with septum. Using a syringe, 0.5mL anhydrous diethyl ether was added. In a separate dry vial, 0.330g bromobenzene and 0.70mL anhydrous diethyl ether were mixed. Immediately after the solution was removed and 0.10mL of it was added to the reaction tube.

Over the next minute the rest of the bromobenzene/ether solution was added carefully so as the reaction does not boil too vigorously. When the boiling slowed down a stir bar was added and the solution was stirred. In a third dry vial, 0.362g of benzophenone was dissolved in 1mL of anhydrous ether. With a dry syringe the solution was added to the Gringard reagent causing a gentle reflux. The vial was rinsed with a small amount of anhydrous ether and was then added to the reaction tube. When the red color disappeared the reaction mixture was cooled in an ice bath. 2mL of 3M HCl was added drop wise while stirring forming a white precipitate. Ether was added to dissolve the precipitate. The aqueous layer was removed and an equal volume of aqueous NaCl solution was added. The aqueous layer was again removed and the ether was dried using anhydrous $CaCl_2$. The ether was removed and it was placed in a tarred reaction tube. The ether was evaporated with N_2. The weight of the crude product was then determined and a TLC was taken. 0.5mL petroleum ether was added and after few minutes the solvent was removed. The remaining residue was the product which was then re-crystallized with 2-propanol. The weight of the pure product was determined and a TLC and IR were performed.

Results

Yield

Compound	Expected Yield	Actual Yield	% Yield
Crude Triphenylmethanol	0.521g	0.355g	68.1%
Pure Triphenylmethanol	0.521g	0.305g	58.5%

TLC

Solvent System: 3:1 Hexane:Ethyl Acetate

Compound	Rf Value
Triphenylmethanol	0.75
Crude Produced Triphenylmethanol	0.75
Triphenylmethanol	0.75
Pure Produced Triphenylmethanol	0.75

IR

Pure Triphenylmethanol: 3469.7cm^{-1}, 3061.0cm^{-1}, cluster of peaks between 1597.8cm^{-1} and 1445.5cm^{-1}

Conclusion and Discussion

After completing the experiment, it was concluded that the reaction did go to completion and the triphenylmethanol formed. The IR scans of the triphenylmethanol showed a large peak at 3469.7cm^{-1} which corresponds to the O-H alcohol peak (3600cm^{-1} to 3200cm^{-1}) found in the molecule. The distinguished peak at 3061.0cm^{-1} represents the sp^3 C found in the final product that contains the hydroxyl group and the three phenyl groups attached to it. The group of peaks found between 1597.8cm^{-1} and 1445.5cm^{-1} represent the presence of a phenyl group in the product, in this case three of them. The fact that no peak exists between the absorbance frequencies of 17258cm^{-1} and 17058cm^{-1} means that no C=O ketone bond exists. A C=O ketone

bond is found in the benzophenone, one of the reactants in this experiment. The lack of that peak in the IR shows that the reaction went to completion.

The TLC performed which compared the product with triphenylmethanol, further proved that the reaction went to completion. The Rf values for the triphenylmethanol found in the lab and the crude and pure triphenylmethanol formed in the reaction were all 0.75. This means that the molecules were the same and the fact that no other spots were found on the TLC plate proves that the product was pure.

The % yield for this reaction was 58.5%. This medium % yield can be attributed to the use of many reaction vessels. When transferring the reaction mixture from one reaction vessel to the next and finally onto the weighing paper, each time a small amount of the product was lost. Since the expected amount of product is so low, even the smallest loss of product is manifested in a big way.

Yield → Water Contamination -2