

The grignard synthesis of triphenylmethanol essay



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The purpose of this lab was to synthesize triphenylmethanol from benzophenone and bromobenzene by the formation of a Grignard compound with the reagents bromobenzene and magnesium metal. The bromobenzene was first transformed into the Grignard compound and was then reacted with the benzophenone to make the final product.

The mixture was then mixed with sulfuric acid and the organic layer was extracted via a separatory funnel. The mixture was then recrystallized from methanol and was allowed to dry and the percent yield, melting point, and the IR was obtained. The mass of the product obtained was 5.45 grams and the percentage yield was determined to be 41.95%. The melting point range obtained from the final product was 89-91°C which was much lower than the literature value of 160-163°C. The peaks from the IR showed the formation of an alcohol group at 3460 wavenumbers and the presence of aromatic rings at 1600 and 1490 wavenumbers which confirms the successful formation of triphenylmethanol.

Introduction: The Grignard reagent is an effective reducing agent that may be used to reduce a ketone to an alcohol. Generally, the Grignard reagent is represented by an alkyl- or aryl-magnesium halide. The nucleophilic Grignard reagent attacks an electrophilic carbon, which results in the formation of a carbon-carbon bond. Forming carbon-carbon bonds are very synthetically useful. The electrophilic carbons found within a carbonyl group are the most likely to be attacked because of their polarity. The Grignard reagent is formed by the reactions between an alkyl or aryl-halide and magnesium. The reaction proceeds via a radical electron transfer. Iodine can also be added to

the reaction. The iodine lies on the magnesium and acts a catalyst to help initiate the reaction.

Scheme 1. Formation of the Grignard reagent. The entire formation of the Grignard reagent takes place in diethyl ether. Diethyl ether prevents oxygen and carbon dioxide from reacting with the Grignard reagent once it is formed. Diethyl ether stabilizes the reagent, because the slightly negative oxygen on each end of the molecule is attracted to the slightly positive magnesium components of the Grignard molecules. Diethyl ether does not react with the Grignard reagent.

Scheme 2. Quenching of the Grignard Grignard reagents must be formed under anhydrous conditions because water will quench the Grignard reagent, rendering it useless for the purpose of our experiment. The Grignard reaction may be done in an environment containing Argon or Nitrogen gas in order to push out all water from the system or to prevent water from entering the reaction flask. Aprotic solvents may also be used in order to keep the nucleophile un-stabilized, and therefore highly reactive. After the Grignard reagent is formed, it reacts with a carbonyl group to form an alkoxide anion. The anion can then be protonated to form the final alcohol product. The complete reaction is as follows:

Scheme 3. Grignard with Aldehyde reaction A dry reflux is also utilized during this experiment. The dry reflux allows for the reaction environment to be almost air free. The use of diethyl ether makes for an anhydrous environment; therefore the reaction flask is free from any unwanted side products. The reflux apparatus is as follows:

Scheme 4. Dry reflux apparatus

Experimental: First a reflux system was set up with a 250 mL round-bottom flask clamped above a stir plate. A water cooled condenser is then attached to a Claisen adapter that is then attached to the round bottom flask. Then a drying tube is attached to a thermometer adapter that is then attached to the top of the condenser. Off of the curved arm of the Claisen adapter, a 250 mL separatory funnel is then attached. 1. 49 grams of magnesium metal turnings were then placed into the round bottom flask and a magnetic stir bar was added to the flask. The magnesium metal was the stirred for several minutes.

Then a small amount of iodine crystal was added to the flask. 40 mL of anhydrous ether was then added via the separatory funnel. 5. 3 mL of bromobenzene and 15 mL of anhydrous ether was then placed into the separatory funnel and was shaken and vented in order to mix the solution. Half of the bromobenzene solution was added first into the round bottom flask and as soon as a color change was observed, the remaining half of the bromobenzene was added drop wise into the round bottom flask. The mixture was then refluxed on a heating mantle for 10 minutes until most of the magnesium has been consumed.

After the initial mixture has refluxed, 9. 11 grams of benzophenone was dissolved in 100 mL of anhydrous ether in a beaker and was then transferred into the separatory on the reflux apparatus. This solution was then added to the Grignard reagent at a drop wise rate while stirring. After the benzophenone was added, the mixture was then refluxed for 15 minutes on

a heating mantle. After the mixture finished refluxing, the flask was then cooled on ice. A sulfuric acid solution was then prepared by pouring 4.5 mL of concentrated H₂SO₄ over 50 grams of ice and then diluted to 75 mL by adding enough tap water to reach 75 mL.

The sulfuric acid solution was then cooled on ice. The Claisen adapter was then removed from the 250 mL round bottom flask and the condenser was then attached to the top of the round bottom flask which was then put on ice. The sulfuric acid solution was then poured through the condenser in order to protonate the product. A stirring rod was then used to dislodge any solid that was stuck to the bottom of the round bottom flask. The solution was then poured into a 500 mL separatory funnel and the layers were allowed to separate. The organic layer was saved and the aqueous layer was then poured back into the separatory funnel.

Another two extractions were performed with 50 mL of ether for each extraction with the returned aqueous layer. The ether layers were then combined and dried over MgSO₄ in an Erlenmeyer flask. The flask was then covered with an inverted beaker until the purification process and allowed to dry for one week. Upon the start of the purification process, the mixture was first filtered and evaporated to dryness on a steam bath. The melt was then cooled on ice to solidify the triphenylmethanol. Methanol was then heated on a steam bath until boiling and enough methanol was added to the flask containing the triphenylmethanol to completely dissolve the solid in the flask.

After completely dissolving the solid, the flask was then filtered with a hot gravity filter in order to purify the solution. After the solution was run through the hot gravity filter, the flask was placed on the windowsill in order to cool down the solution. After the flask reached room temperature, the flask was cooled on ice in order to make sure as much of the product was solidified as possible. The precipitate was then collected over a vacuum filtration system and the final product was allowed to dry for one week. The IR and the melting point were then obtained using a Mel-Temp apparatus and a IR spectrometer.